Harding Lawson Associates

Rocky Mountain Arsenal Information Center Commerce City, Colorado

This document is intended to comply with the National Environmental Policy Act of 1969.

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY *(Leave blank)*	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	08/00/89	

4. TITLE AND SUBTITLE
OFFPOST OPERABLE UNIT, DRAFT FINAL QUALITY ASSURANCE PLAN

5. FUNDING NUMBERS
DAAA15 88 D 0021/0001

6. AUTHOR(S)

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
HARDING LAWSON ASSOCIATES
DENVER, CO

8. PERFORMING ORGANIZATION REPORT NUMBER
89268R01

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
ROCKY MOUNTAIN ARSENAL (CO.). PMRMA
COMMERCE CITY, CO

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT
APPROVED FOR PUBLIC RELEASE; DISTRIBUTION IS UNLIMITED

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

THIS QUALITY ASSURANCE PLAN FOCUSES ON EFFORTS THAT GENERATE ANALYTICAL CHEMISTRY DATA. IT SETS FORTH STANDARD PRACTICES THAT WILL BE USED DURING THE PERFORMANCE OF OFF-POST ACTIVITIES. SECTIONS OF THIS PLAN PRESENT INFORMATION ON:

1. LABORATORY CERTIFICATION
2. SAMPLE COLLECTION AND MANAGEMENT
3. ANALYSES
4. SYSTEM CONTROLS
5. INSTRUMENT MAINTENANCE
6. RECORDKEEPING
7. REPORTS.

14. SUBJECT TERMS	15. NUMBER OF PAGES
SAMPLING, EQUIPMENT	16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED			

TECHNICAL SUPPORT FOR ROCKY MOUNTAIN ARSENAL

Offpost Operable Unit
Draft Final Quality Assurance Plan

August 1989
Contract Number DAAA15-88-D-0021/0001
RIFS1

PREPARED BY:

HARDING LAWSON ASSOCIATES

PREPARED FOR:

PROGRAM MANAGER FOR
ROCKY MOUNTAIN ARSENAL

THIS DOCUMENT IS INTENDED TO COMPLY WITH THE NATIONAL ENVIRONMENTAL POLICY ACT OF 1969.

THE INFORMATION AND CONCLUSIONS PRESENTED IN THIS REPORT REPRESENT THE OFFICIAL POSITION OF THE DEPARTMENT OF THE ARMY UNLESS EXPRESSLY MODIFIED BY A SUBSEQUENT DOCUMENT. THIS REPORT CONSTITUTES THE RELEVANT PORTION OF THE ADMINISTRATION RECORD FOR THIS CERCLA OPERABLE UNIT.

TABLE OF CONTENTS

TABLE OF CONTENTS
(Continued)

TABLE OF CONTENTS
(Continued)

LIST OF TABLES

LIST OF FIGURES

1.0 INTRODUCTION

This Quality Assurance (QA) Plan has been prepared by Harding Lawson Associates (HLA) as deliverable A006, a requirement under Delivery Order 0001 (RIFS1) of Contract DAAA15-88-D-0021 between HLA and the U.S. Army Armament, Munitions and Chemical Command. This document, submitted as a comparison document to the Interim Response Action (IRA) Work Plan (A005), provides guidance for data collection activities to be performed as part of the Offpost Operable Unit Investigation being conducted at Rocky Mountain Arsenal (RMA). This QA Plan was developed in accordance with QA procedures set forth in the PMRMA Chemical QA Plan (Version 1.0, July 1989) and with guidance from the QA plans of contractor laboratories.

The purpose of this QA Plan is to document HLA's responsibilities in coordinating implementation of the RMA Chemical QA Plan. This QA Plan sets forth standard practices that will be utilized by HLA and its subcontractors during performance of offpost activities. This QA Plan was designed to:

1. Provide a consistent framework for generation of analytical data
2. Address the quality of analytical systems used in performing data collection activities
3. Set forth procedures that demonstrate that analytical systems are in control
4. Set forth procedures that limit the effect of non-laboratory activities on analytical data quality
5. Set forth recordkeeping procedures commensurate with project data uses
6. Provide for generation and documentation of data that are of the highest technical merit and utility

Implementation of this QA Plan will help monitor and control the quality of data and provide a reliable foundation on which to base program decisions.

1.1 SCOPE

The scope of this QA Plan addresses both analytical and field measurement results. When properly implemented, this QA Plan will generate documentation that field and analytical

measurements have been obtained under controlled conditions. QA, as used in this document, includes the system of checks and reports used to monitor activities related to data quality. QA refers to all documentation related to the traceability, completeness, and security of analytical or field documents. Quality control (QC), as used in this document, includes the specific actions taken to demonstrate that system performance is maintained at the levels specified by PMRMA. To ensure accuracy, precision, and comparability of results, QC activities are conducted and documented within a QA system.

1.2 APPLICATION

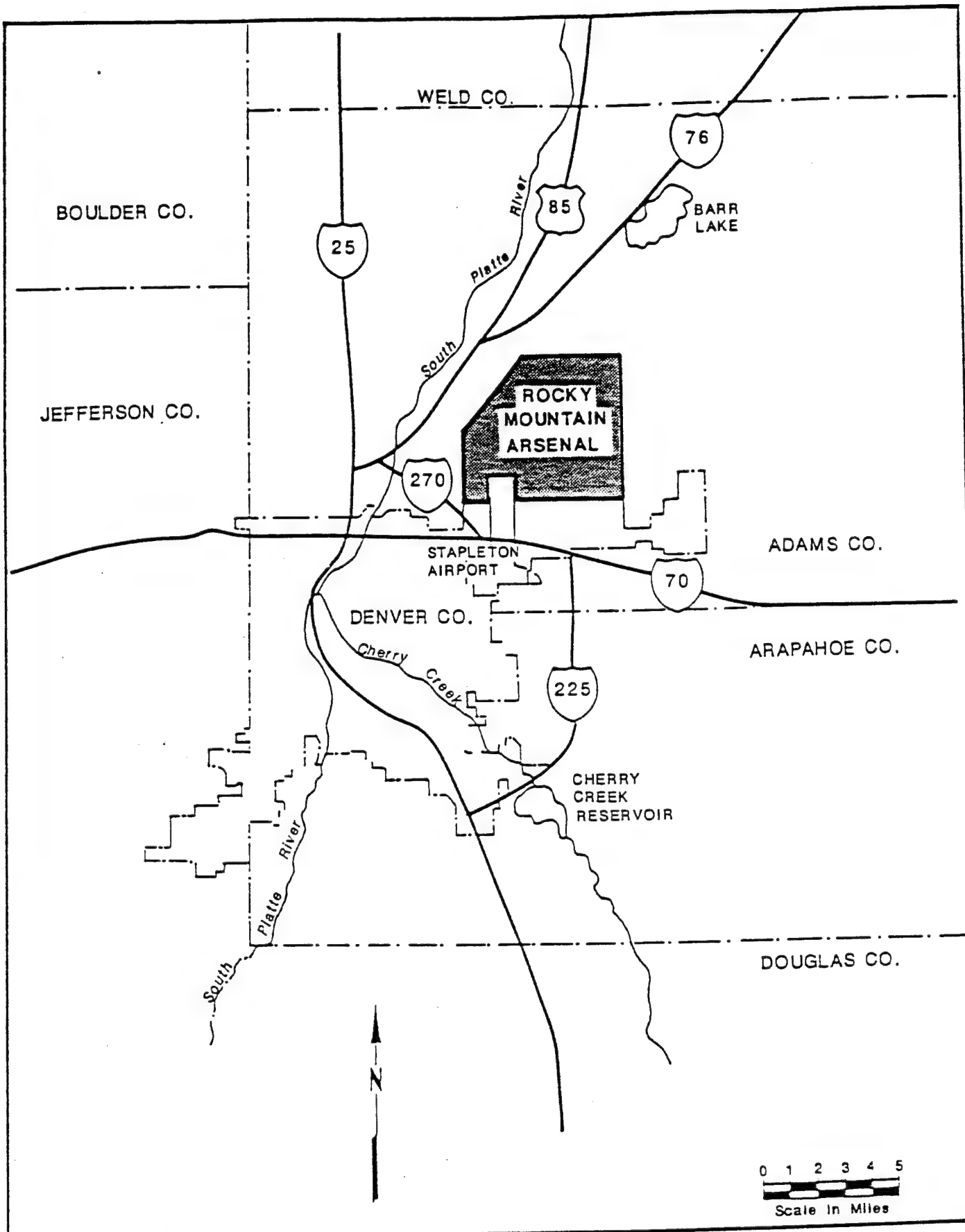
This QA Plan focuses on activities that generate analytical chemistry data, including aspects of field sampling activities that may affect the chemical integrity of environmental samples. Specific instructions for sampling and chemical analysis of environmental media are provided. This document also describes chain-of-custody procedures, QA of all computer-related activities, and QA of final results calculations.

2.0 PROJECT DESCRIPTION

RMA occupies 27 square miles in southern Adams County, Colorado, and lies within the Denver metropolitan area (Figure 2.1). Since 1942, RMA has been a site for the manufacture and demilitarization of chemical and incendiary munitions and the manufacture of industrial chemicals, primarily pesticides and herbicides. Past industrial activities and waste disposal practices at RMA have resulted in the release of chemical substances to the environment. Activities to investigate the nature and extent of chemical constituents from RMA were initiated in the 1970s and are still in progress. Because of the nature of these releases, the RMA site was added to the National Priorities List (NPL) and was therefore subject to compliance with the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA), also known as Superfund.

Offpost ground-water contamination was first suspected in 1954, when owners of property northwest of RMA reported crop losses following irrigation with shallow ground water. From 1956 to 1974, numerous small-scale investigative studies were performed. In 1974, diisopropyl-methylphosphonate (DIMP) and dicyclopentadiene (DCPD) were detected in offpost surface water, and the Colorado Department of Health (CDH) issued three administrative orders to cease and desist offpost releases of hazardous substances.

In response to regulatory requirements, larger-scale environmental investigations were initiated at RMA. These investigations identified approximately 130 potential sources of soil contamination, probable contaminant migration pathways, and areas of ground-water contamination. Following the assessment of onpost ground-water contaminant plumes, three discrete ground-water extraction and treatment systems were designed and installed to prevent offpost discharge of contaminated ground water. These systems (which are currently in operation) include the Northwest Boundary Control System (NWBCS) and the North Boundary Control System (NBCS), both of which were installed and operated by the U.S. Army, and the Irondale Control System (ICS), which was installed and operated by Shell.



Prepared for:
Program Manager for
Rocky Mountain Arsenal

Commerce City, Colorado

Figure 2.1
LOCATION MAP OF
ROCKY MOUNTAIN ARSENAL

20000.140.10

A RI/FS program was initiated at RMA in 1984. This program included regional and localized investigations of soil, ground water, surface water, biota, and air in onpost and offpost areas.

Objectives of previous offpost RI/FS activities have included:

- Identification of compounds attributable to RMA releases
- Evaluation of the extent of ground-water contamination
- Evaluation of contaminant transport pathways
- Identification of areas of potential public exposure
- Generation of a comprehensive water-quality data base for use in the assessments listed above
- Assembly, evaluation, and selection of a preferred remedy to mitigate the threat posed by offpost contamination to humans and the environment

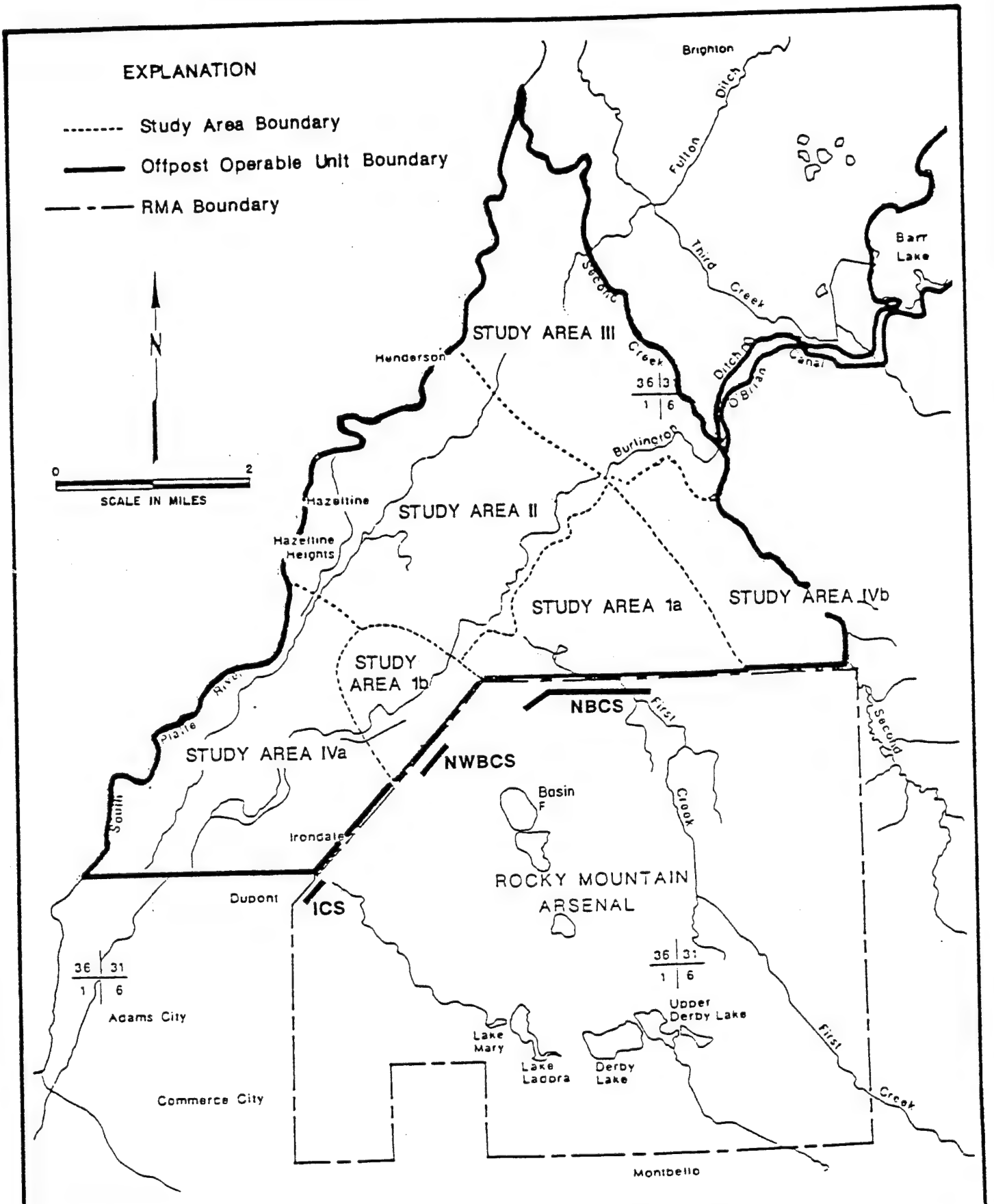
2.1 DESCRIPTION OF STUDY AREA

The offpost area included in the RIFS1 studies is located north and northwest of the RMA, as shown in Figure 2.2. This area has been subdivided into smaller areas on the basis of the extent of contamination and on common media and contaminants of concern. Within the entire RIFS1 area, two primary ground-water contaminant migration pathways have been identified, one in the unconfined aquifer directly north of RMA's northern boundary and one in the NBCS.

2.2 QA/QC OBJECTIVES

The following QA/QC objectives were developed to comply with the PMRMA Chemical QA Plan guidelines and the specific objectives and goals of the RIFS1 program. Achieving these objectives is critical to successful completion of the field and analytical program objectives. The QA/QC objectives listed below will apply to all field and analytical operations:

- Evaluate the technical utility of field and analytical results
- Document and control procedures for sample collection, preservation, and handling
- Establish external QC sampling schedules and validation review procedures to adequately address analytical precision, accuracy, representativeness, completeness, and comparability



Prepared for:
Program Manager for
Rocky Mountain Arsenal

Commerce City, Colorado

Figure 2.2
OFFPOST STUDY AREAS

20000,140.10

- Set guidelines for data tracking and validation requirements to quickly determine data deficiencies and quality
- Clearly state corrective action procedures and documentation to address ongoing analytical and field-related problems
- Provide audit procedures for evaluation of field and laboratory operations to ensure that all project operations and analyses are conducted in a manner consistent with the PMRMA Chemical QA Plan guidelines
- Identify overall project and QA personnel, including key laboratory QA and project management personnel
- Tabulate the responsibilities of key project personnel
- Compile project and laboratory personnel qualification information to assure that appropriate personnel are identified to accomplish project QA objectives
- Confirm that laboratory facilities, equipment, and services are adequate to achieve QA objectives

2.3 IMPLEMENTATION OF THE PMRMA CHEMICAL QA PLAN AND QA/QC PROJECT OBJECTIVES

HLA's Quality Assurance Coordinator (QAC), under the guidance of HLA's Program QA Manager and PMRMA's Project QA Officer will conduct and oversee implementation of all PMRMA Chemical QA Plan requirements. QA/QC tracking forms or sign-off forms will be utilized and reviewed by the QAC to establish that samples or data have been collected, logged, transported, analyzed, transcribed, validated, group checked, and transmitted to PMRMA. The details for achieving each of these sample and data processing activities are detailed in this QA Plan. Guidelines and procedures are described as they appear in the PMRMA Chemical QA Plan or as appropriate to meet specific goals. The QAC will prepare and submit periodic reports to management concerning field, laboratory, and data management QC issues.

Audits will be conducted to review field activities and procedures for laboratory record-keeping, data validation, and data management and to assess whether field and laboratory practices comply with the guidelines established in the PMRMA Chemical QA Plan and procedures established in this QA Plan. Audits, which will be performed both announced and unannounced, will be used to evaluate completeness and comparability of analytical data. Audit

reports will be issued to project management following each audit. Corrective action will be fully described in the audit reports, with a timetable established for laboratory compliance with the required action. The audit reports will present the nature of the required corrective action and will list the appropriate documentation required to demonstrate that corrective action has been implemented. Upon receipt of this documentation, the QAC will convey in writing to the Program QA Manager that the appropriate corrective action has been implemented and documented.

Audits will be used to evaluate and train project personnel in proper operational protocol required by PMRMA and this QA Plan. The QAC and Laboratory QA Coordinator (LQAC) may issue stop-work orders to laboratory section supervisors if an out-of-control situation is found to exist. When a stop-work order has been issued, work will not be resumed until corrective action is performed and the stop-work order is rescinded by telephone or written communication. The order to resume work can be issued only by the party responsible for the original stop-work order.

Analyst and laboratory section supervisors are responsible for identifying out-of-control situations that exist or may potentially occur and informing the LQAC or the QAC that corrective action is required. Failure to identify analytical problems prior to submitting final results may result in a stop-work order, re-analyses and re-sampling, or both. The cost of re-analysis and/or resampling may be assessed against the laboratory if this work be deemed the result of a laboratory oversight or negligence.

3.0 PROJECT ORGANIZATION AND RESPONSIBILITY

To achieve the overall objectives of the RIFS1 program, HLA has formed a functional organization network that will maximize day-to-day operational efficiency. This network has been established to provide consistent technical direction while simultaneously providing effective administrative management for the complex, multidisciplinary work elements. Figure 3.1 shows a diagram of HLA's functional organization for the RIFS1 program.

As a contractor to PMRMA, HLA will be responsible for independent review of all project-related sampling and analysis activities. Ultimately, PMRMA is responsible for the quality of data collected in support of PMRMA projects. This responsibility is delegated to HLA through PMRMA and individual project officers (Figure 3.2).

Analytical data and field sampling efforts will be evaluated by HLA in accordance with the requirements stated in the PMRMA Chemical QA Plan. The USATHAMA Analytical Branch, assisted by the USATHAMA Central QA Laboratory, will also maintain an active, ongoing system of QA. Project subcontract laboratories will maintain a QC program consistent with PMRMA Chemical QA Plan guidelines and will provide ongoing QA information to PMRMA and HLA for review. Laboratories that will perform analyses for RIFS1 will do so using only current PMRMA- or USATHAMA-certified analytical methods and related reporting limits.

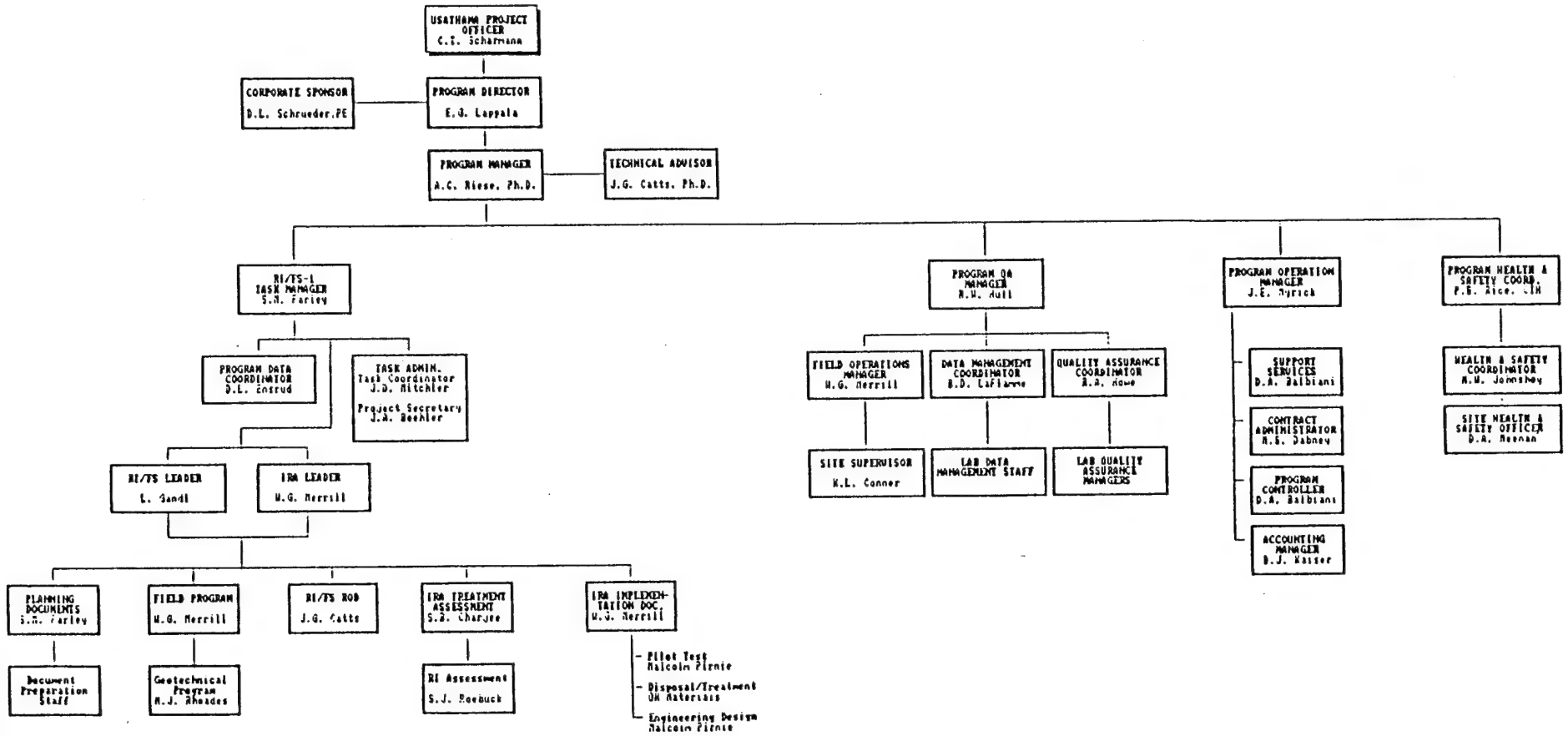
3.1 PROJECT RESPONSIBILITIES

The following section describes project responsibilities of PMRMA, HLA, and subcontractors.

PMRMA:

Project Officer (PMRMA) - Charles Scharmann

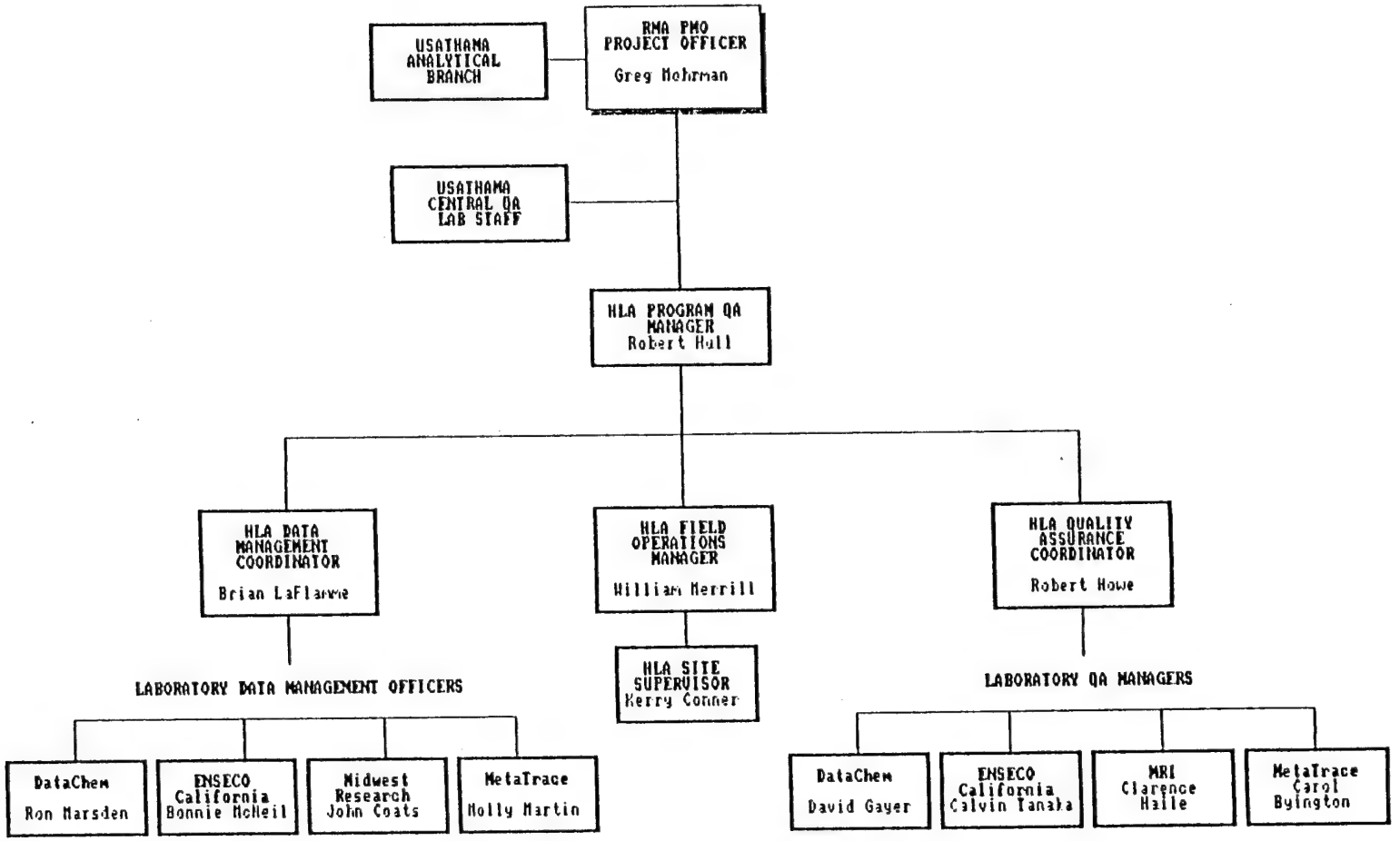
- Principal contact between PMRMA and the prime contractor (HLA)
- Review and coordinate ongoing project activities



Prepared for:
U.S. Army Program Manager's Office
For Rocky Mountain Arsenal
Commerce City, Colorado

Figure 3.1
FUNCTIONAL ORGANIZATION RMA RIFS1

20000.140.10



Prepared for:
U.S. Army Program Manager's Office
For Rocky Mountain Arsenal
Aberdeen Proving Ground, Maryland

Figure 3.2
QA ORGANIZATION RMA RIFS1

7B

20000.

Project QA Officer (PMRMA) - Greg Mohrman

- Principal contact between PMRMA and subcontractor laboratories
- Monitor effective implementation of PMRMA Chemical QA Plan
- Submit requests to the USATHAMA Analytical Branch to supply analytical reference materials to subcontractor laboratories
- Provide formal notification to the QAC of unapproved deviations from the PMRMA Chemical QA Plan or this QA Plan
- Promptly submit QC charts from subcontractor laboratories on a weekly basis
- Provide HLA with a formal list of applicable analytes for each task or activity
- Inform PMRMA of difficulties encountered by subcontractor laboratories in implementing this QA Plan
- Provide subcontractor laboratory certification documentation to HLA
- Notify subcontractor laboratories of method/analyte certification status

Central QA Laboratory (USATHAMA) - Staff

- Maintain the analytical reference material repository
- Provide analytical reference materials and support documentation to subcontractor laboratories
- Provide support to the PMRMA Project Officer in QA/QC areas

HLA - Program Level:

Program Director - Eric G. Lappala

- Responsible for overall design, direction, and implementation of the program
- Guide the approach to problem resolution
- Maintain program integrity

Corporate Sponsor - Donald L. Schreuder

- Provide corporate review of and commitment to the allocation of resources
- Resolve program problems, as needed

Program Manager - Arthur C. Riese, Ph.D.

- Responsible for program administration
- Supervise Task Managers
- Primary liaison between PMRMA and HLA
- Assure adherence to program schedule and budgets
- Control of overall program direction, coordination, technical consistency, safety, and scientific merit

Technical Advisor - John G. Catts, Ph.D.

- Responsible for overall technical direction of the program
- Secondary point of contact for PMRMA and regulatory agencies
- Provide technical review of all project deliverables
- Communicate with technical consultants regarding special concerns
- Review QA/QC procedures

Program QA Manager - Robert W. Hull

- Evaluate QA/QC requirements and deliverables, as necessary or appropriate
- Ensure consistent QA/QC procedures for program activities

Program Health and Safety Coordinator - Peter B. Rice, CIH

- Ensure that individual Health and Safety Plans are consistent with other task plans
- Evaluate health and safety standards and procedures

Program Operations Manager - John E. Myrick

- Administrator RMA program in an efficient manner
- Advise administrative managers, including:
    - Contract Administrator - Michael S. Dabney
    - Program Controller - Daniel A. Balbiani
    - Accounting Manager - Donna J. Kaiser

- Project Coordinator - John D. Mitchler
- Support Services Manager - Daniel A. Balbiani

- Coordinate use of HLA resources
- Oversee administration of contracts

HLA - Task Level:

Task Manager - Stephen M. Farley

- Control overall task direction, coordination, technical consistency, and contract requirements
- Plan and approve schedules and budgets
- Supervise project teams, work element leaders, and technical staff
- Primary liaison between HLA and PMRMA
- Responsible for development of all task deliverables
- Ensure that QA/QC recommendations and corrective actions are implemented

QA Coordinator - Robert A. Howe

- Ensure, oversee, and audit analytical procedures being implemented to meet project and QA Plan objectives
- Document and inform Task Manager and LQAC of nonconformance of laboratory analytical parameters with QA Plan requirements
- Suggest and schedule corrective action procedures to rectify out-of-control situations
- Request analytical reference materials from the USATHAMA Central QA Laboratory
- Audit records, field procedures, logs, laboratory procedures, and project plans and ensure that analytical results are stored in a secure area readily retrievable by project personnel
- Distribute all standard procedures and project plans to laboratory personnel
- Communicate with laboratory personnel the appropriate analytical sample lot size, QC sample percentages, and procedures for evaluating acceptable analytical performance
- Perform unannounced inspections of sampling procedures for each sampling media, including ground water, surface water, soil, sediment, biota, air, and waste, during the first sampling event for each media

- Recommend revised performance objectives for the RIFS1 Work Plan and QA Plan if those objectives are determined to have been adversely affected by faulty sampling procedures

- Cross check chain-of-custody records and lot designations for investigative samples and control samples for correctness and size

- Review and document the precision, accuracy, respresentativeness, comparability, and completeness of the data

- Audit laboratory procedures for preparing QC samples, maintaining control charts, documenting corrective action, and completing all log books, as required by PMRMA and this QA Plan

- Report control chart results to the PMRMA Project Officer on a weekly or case basis

- Maintain awareness of laboratory conditions and scheduled sample loads

- Coordinate activities with the Laboratory QA Coordinators

Data Management Coordinator - Brian D. LaFlamme

- Manage the Installation Restoration Data Management System (IRDMS) and all supporting subroutines or programs to support data retrieval and reduction efforts

- Receive and transfer data between subcontractor laboratories and the PMRMA data management group

- Communicate with and support the QAC in identifying inconsistency in data reporting by laboratory data management personnel

- Inform the QAC of samples that are past due for reporting or extraction, using the automated sample tracking system

- Correct all results for method accuracy and precision

Field Operations Manager - William G. Merrill

- Responsible for all field activities, including QA of field procedures

- Review field data

- Prepare and review field reports

- Recommend appropriate corrective action procedures

- Communicate field objectives to the Site Supervisor

Site Supervisor - Scott Wibby

- Conduct field sampling program in accordance with appropriate sampling plans
- Prepare reports on field activities and results
- Advise Field Operations Manager of field-related QA/QC problems or inconsistencies
- Coordinate field staff activities

Health and Safety Coordinator - Marcus W. Johnshoy, CIH

- Develop Health and Safety Plan
- Provide health and safety training for all task team members
- Inform field personnel and Site Health and Safety Officer of specific safety requirements
- Provide field personnel with written documentation of field safety procedures

Site Health and Safety Officer - D. Anita Meenan

- Supervise field safety procedures
- Provide field safety equipment to field personnel
- Report to Health and Safety Coordinator any inconsistencies between the Health and Safety Plan and site implementation safety practices
- Setup and maintenance of HLA computer and data management systems
- Additional responsibility detailed in RUP

Laboratory Organization

Laboratory QA Coordinators (LQACs)

DataChem - David Gayer

Enesco California - Calvin Tanaka

Midwest Research Institute - Clarence Haile

MetaTrace, Inc. - Carol Byington

- Liaison with HLA QAC and PMRMA Project Officer
- Monitor laboratory workloads and ensure availability of resources
- Analytical report preparation

- Supervise in-house chain of custody
- Periodically review and inspect all laboratory project activities
- Implement the PMRMA Chemical QA Plan and this QA Plan
- Support QAC efforts
- Provide HLA and PMRMA with the required method documentation and certification data prior to the analysis of field samples
- Ensure that subsampling and handling procedures are technically sound and in compliance with historically defined RMA protocol
- Maintain and oversee the quality of laboratory materials such as reagents and chemical supplies
- Document and communicate the results and corrective action procedures to the QAC

Laboratory Data Management Officers:

DataChem - Ron Marsden

Enesco California - Bonnie McNeil

Midwest Research Institute - John Coats

MetaTrace - Molly Martin

- Responsible for compilation and entry of lot analysis data
- Responsible for control chart generation and delivery to HLA on a weekly or demand basis
- Transfer data electronically or on computer disk to HLA
- Correct data entry errors indicated after HLA validation process
- Inform LQAC of data deficiencies or inconsistencies prior to delivery to HLA

4.0 LABORATORY CERTIFICATION

Contractor laboratories must demonstrate the ability to perform method analysis necessary to meet task objectives. Standard analytical methods to be used for analysis of environmental samples will be provided by PMRMA. Laboratories must provide PMRMA and HLA with the current status and code designations for all certified project-related methodologies.

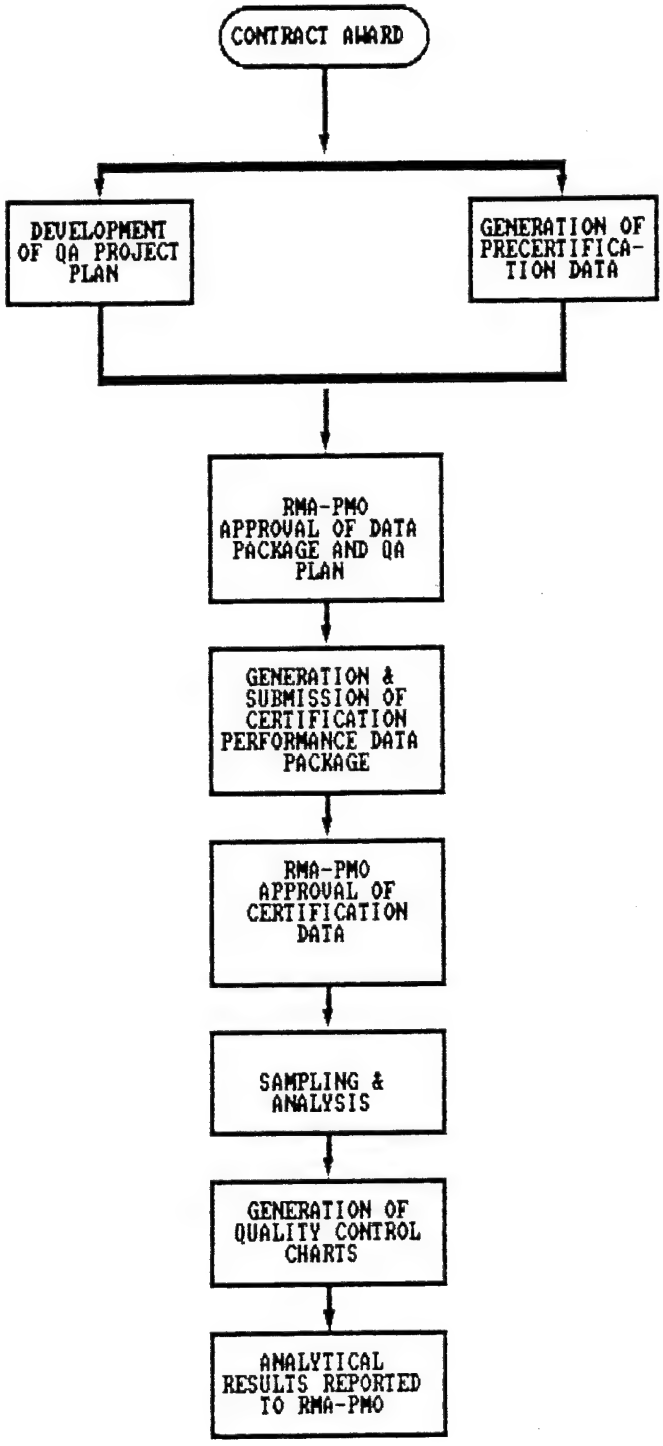
Laboratory certification is a two-phase process involving an initial submission of data from precertification calibration standards followed by submission of data from certification performance samples. Precertification calibration standards are presented as a data package to establish instrument reliability and precision. Certification performance samples are then analyzed by the contractor laboratory, and the results are submitted to PMRMA as confirmation of the laboratory's proficiency in conducting a given analysis. After certification approval, the laboratory will be provided with a unique method number to be used when reporting analytical project data. The sequence for certification activities is shown in Figure 4.1.

4.1 ANALYTICAL METHODS

Analytical methods to be certified must include written instructions describing the analytes, sample type, sample preparation, reagents required, calibration, and computations that are an integral part of the method. Certified methods may not be altered unless approved by PMRMA. The standard format for documentation of analytical methods is provided in the PMRMA Chemical QA Plan and in Appendix A of this document.

4.1.1 Standard Methods

To provide a common point of reference, PMRMA prescribes the use of standard methods for commonly encountered analytes. These methods are sufficiently general to be used by most laboratories, yet specify all critical elements. The methods published by the U.S. Environmental Protection Agency (EPA), the American Society for Testing and Materials (ASTM), or past USATHAMA methods may be used as a basis for certified methods if they specify techniques for



Prepared for:
U.S. Army Program Manager's Office
For Rocky Mountain Arsenal
Commerce City, Colorado

Figure 4.1
SEQUENCE FOR CERTIFICATION ACTIVITIES

20000,140.10

sample preparation, calibration frequency, calibration check acceptance criteria, standard stock solution preparation, and QC sample preparation.

4.1.2 Methods Not Requiring Certification

Various analytical methods or measurements do not require certification because of the nature of the measurement or the intended use of the data. PMRMA does not supply standardized methodologies for such procedures or measurements. For this task, the analytes or parameters listed below will be measured by EPA standard test method procedures. Laboratories will be supplied with the method to be used or will be requested to supply detailed information describing the exact procedures to be used. The following parameters or tests do not require certification:

- Sulfide
- Ammonia nitrogen
- Biological Oxygen Demand (BOD)
- Temperature
- Conductivity
- pH
- Oil and grease
- Hardness
- Asbestos
- Alkalinity, carbonate, bicarbonate, hydroxide
- Total Organic Carbon (TOC)
- Chemical Oxygen Demand (COD)
- Total Dissolved Solids (TDS)
- Total Suspended Solids (TSS)

Methods utilized as general indicators may not require certification, but when any of the parameters listed above are deemed critical for task decisions, certification may be required.

4.2 PRECERTIFICATION

A calibration curve will be constructed for the proposed project analytes at concentrations similar to those expected to be found in the investigative samples. Each standard will be prepared as a concentrate and run in duplicate. Extensions to the required minimum working method range should include at least a 10 percent extension for inorganic analyses of waters and a 25 percent extension for all other methods. Standards will be prepared by the laboratory, and calibration results will be tabulated and graphed to show instrument response versus concentration. Precertification calibration standard curves will then be evaluated by the laboratory for linearity (lack of fit [LOF]) and accuracy (zero intercept [ZI]). Deviations from linearity will be reviewed and accepted or rejected by PMRMA prior to continuation of method certification. A more complete description of these tests is provided in the PMRMA Chemical QA Plan. Appendix B of this QA Plan provides an example of these precertification test results. Accuracy data will be incorporated into the method data base and will be utilized to correct investigative sample results. Approval of precertification data packages is required before certification can continue.

Calibration check standards must also be obtained from EPA or another commercial source and analyzed to verify the accuracy of laboratory prepared calibration standards. Analytical results for these calibration check standards and the EPA/commercial true concentration documentation must be submitted with the precertification calibration curve data. Results for calibration check standards must comply with the acceptability limits defined by the originator.

4.3 CERTIFICATION

Each contractor laboratory, after completing the precertification calibration curves for each method, must demonstrate proficiency in conducting chemical analyses by analyzing spiked standard samples (certification samples). The certification samples will be prepared by the contractor laboratory from the same standard reference materials used to spike standards during precertification analyses. Standard water samples will be prepared by adding a known quantity of

target analyte to a known volume of Type I Water (See Table 4.1) for inorganics and to ASTM Type II Water for organics. Type II Water will be prepared by the contractor laboratory following ASTM guidelines. Standards will be spiked into the aqueous or reagent media appropriate for the method. Solid sample standards are prepared by adding a known quantity of each target analyte to a known weight of blank soil provided by PMRMA. The amount of blank soil to be used will be specified by the method utilized to perform the analyses. PMRMA will provide the laboratory with a sufficient quantity of standard soil to meet certification requirements.

Standard biota samples used in the certification process will be National Bureau of Standards (NBS) bovine liver for animal tissues and citrus leaves for plant and foliage analysis. Biota certification using tissues and citrus leaves will be performed in the same manner as that for a soil or sediment method.

4.3.1 Certification Analyses

Prior to the initiation of certification analyses, a multilevel calibration must be performed. Methods will be certified and analytical data will be utilized in four different ways. Specific project requirements will dictate the level of analytical precision required. Certifications are designated as Class 1, Class 1A, Class 1B, or Class 2 and are used to characterize laboratory performance and the technical utility of analytical results. Class 1A certification is reserved for gas chromatography/mass spectrometry (GC/MS) methods. Class 1 and 1B methods are reserved for low throughput methods or non-GC/MS methods. Class 2 methods are those from which only qualitative results may be obtained.

Class 1 and Class 1B results can be reliably reported to include three significant figures. Class 1A certified results can be reported to include two significant figures. Non-certified Class 1A analytes (Tentatively Identified Compounds or special compounds of interest) may be reported to include only one significant figure. Class 2 method results can be used to screen for the presence or absence of chemical constituents relative to a target reporting limit (TRL) or desired

Table 4.1: Criteria for ASTM Water Types

Grade of Water	Maximum Total Matter (mg/l)	Maximum Electrical Conductivity at 25C (umho/cm)	Minimum Electrical Resistivity at 25C (MΩcm)	Minimum Color Retention Time of $KMnO_4$ (min)
Type I	0.1	0.06	16.67	60
Type II*	0.1	1.0	1.0	60

*100 mg/l Sulfate and Chloride added. The following preparation is provided:

(1) Weigh 1.48 g of reagent grade anhydrous sodium sulfate into a 1-liter volumetric flask and dilute to mark with ASTM Type II water.

(2) Weigh 1.65 g of reagent grade anhydrous sodium chloride into a 1-liter volumetric flask and dilute to mark with ASTM Type II water.

(3) Transfer 100 ml of each solution prepared in (1) and (2) into a 1-liter flask and dilute to volume with ASTM Type II water.

action level (DAL) only. Results of Class 2 methods are reported as "less than," "equal to," or "greater than" TRL or DAL.

Concentration Range

For each analyte, a TRL must be selected prior to analysis. When possible, PMRMA will define the TRL for a method analyte(s) based on the lowest method detection limits technologically obtainable. For each analyte/method combination, the contractor laboratory must discuss the anticipated project sample concentrations and their proposed working range of the method to be employed. The working range of all classes of certifiable methods must include the TRL and, except for Class 2 methods, extend to a minimum level of concentration of 10 times the TRL. Extensions of any method's upper limit of performance is defined only by instrument performance and procedural limitations. Each order of magnitude increase above the minimum testing range must include three additional calibration standard concentrations for Class 1 and 1B methods. For Class 1A methods, two additional concentrations at multiples of the TRL must also be inserted.

Class 1 and Class 1B Certification:

Certification analyses will be conducted over a four-day period by performing a minimum of one performance standard analysis at each concentration level per day (See Table 4.2). The required analyses will be conducted on consecutive days and will include the entire analytical process from extraction to instrument response. Analysis includes performance of the entire method, including spiking samples and sample preparation.

Class 1A Certification

Two standard samples at each concentration level will be analyzed on a single day. Standard stocks used in certification will be independently prepared from separate master stock solutions. Certified reporting limits (CRLs) and method accuracy will be generated by the contractor laboratory using standards prepared at concentrations shown in Table 4.2.

Table 4.2: Numbers and Concentrations of Certification Samples

CLASS 1/CLASS 1B

Minimum Testing Range (MTR): 24 Certification Samples
Blank, 0.5, 1, 2, 5, & 10 TRL (4 Days)

MTR + 1 Order of Magnitude Extension: 36 Certification Samples
Blank, 0.5, 1, 2, 5, 10, 20, 50, & 100 TRL (4 Days)

MTR + 2 Orders of Magnitude Extension: 48 Certification Samples
Blank, 0.5, 1, 2, 5, 10, 20, 50, 100, 200, 500, & 1000 TRL (4 Days)

CLASS 1A

Minimum Testing Range (MTR): 8 Certification Samples
Blank, 0.5, 2, & 10 TRL (Duplicate)

MTR + 1 Order of Magnitude Extension: 12 Certification Samples
Blank, 0.5, 2, 10, 50, & 200 TRL (Duplicate)

MTR + 2 Orders of Magnitude Extension: 16 Certification Samples
Blank, 0.5, 2, 10, 50, 200, 500, & 2000 TRL (Duplicate)

CLASS 2

Minimum Testing Range: 8 Certification Samples
Blank, 1 TRL (Quadruplicate)

Class 2 Certification

A minimum of four standards at the concentration level equivalent to the TRL and four blanks will be analyzed in a single day using the complete analytical method. The Rank Sum Test will then be applied (Table 4.3), and results must not sum to a value greater than 10 using this test procedure. If analytical tests rank sum is greater than 10, additional samples (2 blanks and 2 standard spikes) may be run to attempt to improve the analytical evaluation data for the method. In this case, the rank sum must not exceed 26. If the method fails, the TRL must be increased until criteria can be met.

Method Development

If project objectives require the use of a nonstandard method to achieve a desired result, development of a method will be conducted by a laboratory certified by PMRMA. Documentation for any proposed method will satisfy the list of deliverables provided in this document and in the EPA guidelines for method equivalency, if applicable. PMRMA must approve the proposed approach and any required change in an analytical approach. Any nonstandard method must be accompanied by the information normally required in standard method precertification and certification performance data packages. PMRMA must review all such performance packages to determine whether they satisfy the required goals for completeness and comprehension. After a method has been approved by PMRMA, a method number will be assigned and the procedures will be issued as a standard analytical method.

4.4 STATISTICAL EVALUATION PROCEDURES FOR CERTIFICATION

This section describes the statistical routines performed by PMRMA software programs on precertification calibration analyses and certification analyses to quantify the technical utility of an analytical method. Methods that fail to meet statistical criteria may be acceptable but must be discussed with PMRMA prior to use.

Table 4.3: Rank Sum Test

Standard Sample	Results*	Rank	Average Rank**
Blank	NN	1	2
Blank	NN	2	2
Blank	NN	3	2
Blank	PP	4	6
Spike	PP	5	6
Spike	PP	6	6
Spike	PP	7	6
Spike	PP	8	6

* NN = Negative; PP = Positive

** Average Rank for Negative Results $= \frac{1 + 2 + 3}{3} = 2$

Average Rank for Positive Results $= \frac{4 + 5 + 6 + 7 + 8}{5} = 6$

Sum of Average Ranks for Blanks $= 2 + 2 + 2 + 6 = 12$

Because the sum of the average ranks of blanks exceeds the criterion of less than or equal to 10, the results are unacceptable.

Test an additional two Blanks and two Spikes:

Standard Sample	Results	Rank	Average Rank***
Blank	NN	1	3
Blank	NN	2	3
Blank	NN	3	3
Blank-New	NN	4	3
Blank-New	NN	5	3
Blank	PP	6	9
Spike	PP	7	9
Spike	PP	8	9
Spike	PP	9	9
Spike	PP	10	9
Spike-New	PP	11	9
Spike-New	PP	12	9

*** Average Rank for Negative Results $= \frac{1 + 2 + 3 + 4 + 5}{5} = 3$

Average Rank for Positive Results $= \frac{6 + 7 + 8 + 9 + 10 + 11 + 12}{7} = 9$

Sum of Average Ranks for Blanks $= 3 + 3 + 3 + 3 + 3 + 9 = 24$

Because the sum of the average ranks of blanks meet the criterion of less than or equal to 26, the certification is acceptable.

Statistical Analysis of Class 1, Class 1A, and Class 1B Performance Certification Data

Results of certification analyses must be statistically evaluated by comparing the reported concentration for the standard spiked sample with the target spiked concentration. Values used for comparison are uncorrected for blank artifacts and surrogate recovery. Statistical calculations should use significant figures appropriate for the method class, as described in Section 4.3.1. Certification results will be evaluated for linearity using LOF analysis and a ZI test. Data collected for statistical evaluation must have been collected under in-control instrument conditions. This condition is defined by response factor for inorganic compounds that are within 10 percent of the mean response factor for surface water and ground water. Organic analyte response factors must be within 25 percent of the mean response factor. Linearity of calibrated results is tested because estimation of method certified reporting limits (CRLs) assumes instrument linearity. Certification analyses are grouped and tested for LOF. Grouped data must pass the LOF test at the 95 percent confidence level.

Certified Reporting Limit

If instrument response is within the acceptable level of linearity as described above, a statistical relationship can be established to define the lowest sample concentration that can be reported with a high degree of analytical confidence. This concentration level is defined as the method CRL for each analyte of interest. CRLs will be determined using 90 percent confidence limits. CRLs are determined by spiking analytes into method blank soil, water, or biota. Each analytical step involved in a method is performed; thus, the results reflect the efficiency of the entire method. This type of method validation tends to be optimistic because it does not account for actual sample matrix effects; however, CRLs are generally higher than instrument detection limits as a result of extraction or other method inefficiencies. Because all steps involved in a method are reflected in the CRLs, standard operating procedures must be followed each time analyses are performed by a certified method.

The statistical formulation of method CRLs is based on three assumptions:

1. The target concentration and analytically determined concentration share a linear relationship.

2. Variance over a tested range of concentration is homogeneously distributed about the least squares linear regression line for the range tested.

3. Analytically determined concentrations are normally distributed.

Utilizing these assumptions, the least squares regression line may be written in the form

$$Y = Y_o + bX$$

Where:

Y = found concentration
Y_o = Y axis (found concentration) intercept
b = slope of the line
X = target concentration

The least squares regression line is then defined for the paired performance data (X, Y) assuming that errors occur only in Y data points. It is now possible to define the slope of the least square regression line for N number of data points by the following equation:

$$b = \frac{N\sum X_i Y_i - \sum X_i \sum Y_i}{N\sum X_i^2 - (\sum X_i)^2}$$

Where:

N = number of data points
X_i = the i-th target concentration
Y_i = the i-th found concentration

Rearranging this equation, the Y intercept is defined by the following equation:

$$\text{Y axis intercept} = Y_o = \frac{\sum Y_i - b\sum X_i}{N}$$

For the 90 percent confidence interval, the upper and lower boundaries of analytical confidence can be determined using the Student's t-test for small populations of analytical results. The following equations are based on a two-tailed data distribution in which N is replaced by N-2 to account for the loss of two degrees of freedom in estimating the fitted line.

The upper confidence limit above the regression line is given by:

$$Y = Y_o + bX + S_{Y.X}\, t \left[1 + \frac{1}{N} + \frac{X_i - \overline{X})^2}{(X_i - \overline{X})^2} \right]^{1/2}$$

The lower confidence limit about the regression line is given by:

$$Y = Y_o + bX + S_{Y.X}\, t \left[1 + \frac{1}{N} + \frac{X_i - \overline{X})^2}{(X_i - \overline{X})^2} \right]^{1/2}$$

Where:

$$S_{xy} = \left[\frac{\sum [Y_i - (\overline{Y} + b(X_i - \overline{X}))]^2}{N-2} \right]^{1/2}$$

Y_o = calculated Y axis intercept

t = Student's t for 2-tailed P = 0.10 and N-2 degrees of freedom

$\overline{X}$ = average of all target concentrations

$\overline{Y}$ = average of all found concentrations

The calculated reporting limit is defined as the value of X corresponding to a point on the lower confidence interval curve where the value of Y equals the value of Y on the upper confidence limit curve at X = 0. The calculated reporting limit will be considered as the method CRL only if one of the tested spike concentrations is at or below the calculated reporting limit. Otherwise, the lowest found spike concentration will be used as the method CRL for the compound tested.

Accuracy

Method accuracy is evaluated using previously described statistical methods applied to the entire data set. Accuracy, or the slope of the least squares linear regression line, is 100 percent when the slope (b) equals 1. Accuracy is defined based on the complete certification data package

unless performance criteria are not met. For Class 1 and 1B certified methods, truncation of the certification is allowed when attempting to meet performance criteria, with the following restrictions:

1. Upper limit of the certification range to be truncated must be above the minimum range requirements.
2. The data set must include the blank and the three lowest concentrations (0.5 TRL, 1 TRL, and 2 TRL).
3. After each truncation, the slope of the least squares linear regression line cannot change by more than 10 percent from the slope for the total data set. If the slope changes by more than 10 percent after dropping a concentration, the calculated reporting limit may not be used as the CRL and further truncation is not acceptable.

The upper limit of the truncated certification data set represents the highest concentration level reportable for the method. Samples must be run below this concentration level unless HLA or PMRMA has approved the release of such qualitative results. Results for the total data set and each truncated data set will be provided in the certification data set. An example of a truncated data set is provided in Appendix B. Truncation of data sets is not allowed for Class 1A certifications.

In addition to the statistical evaluations described above, other statistical characteristics presented below, including standard deviation, inaccuracy, and imprecision, will be provided with each certification data set (except Class 2 methods, for which accuracy cannot be defined). These calculations are also performed by PMRMA software and are provided in Appendix B.

Standard Deviation

For Class 1, Class 1A, and Class 1B certification, the standard deviation, S, will be calculated at each target concentration according to:

$$\text{Standard Deviation} = S = \left[\frac{\sum Y_i^2 - \frac{(\sum Y_i)^2}{N}}{N - 1} \right]^{1/2}$$

Where:

Y_i = the found concentration
N = total number of Y values at each target concentration

Inaccuracy

For Class 1, Class 1A, and Class 1B certification, the percent inaccuracy will be calculated at each target concentration according to:

$$\text{Percent Inaccuracy} = \frac{\overline{Y} - X}{X} (100)$$

Where:

X = target concentration

$\overline{Y}$ = average found concentration at the target concentration

Imprecision

For Class 1, Class 1A, and Class 1B certification, the percent imprecision will be calculated at each target concentration according to:

$$\text{Percent Imprecision} = \frac{S}{\overline{Y}} (100)$$

Where:

S = standard deviation

$\overline{Y}$ = average found concentration at the particular target concentration

Documentation

Upon completion of either precertification or certification, performance testing documentation consistent with the formats described in this QA Plan and in the PMRMA Chemical QA Plan must be delivered to the PMRMA Project QA Officer.

5.0 SAMPLE COLLECTION AND MANAGEMENT

Field activities for the RIFS1 program are designed to provide data adequate to assess the nature and extent of chemicals in various media at the site. Various environmental media will be sampled, including tap water, surface water, ground water, soil, sediment, and biota. Sample collection activities for these media have been developed so that the sampling techniques used and the number and location of samples collected are representative of site conditions. Sampling techniques to be utilized are detailed in the RIFS1 Field Operations Plan (FOP).

Sampling activities are documentated to verify that sample integrity is maintained during sample collection, transportation, and storage prior to analyses. Documentation in the sample management program provides a record of procedures used in sample collection and analysis. Sample collection procedures and management are discussed in the following sections.

5.1 SAMPLE COLLECTION

Sample collection procedures to be utilized during the field investigation have been developed to meet the minimum QA requirements described in the PMRMA Chemical QA Plan. The required sample containers/volume, preservation methods, holding times, and general QA procedures for sample collection are described in the following sections.

5.1.1 Sample Containers

Six different types of sample containers will be used to store and transport samples for all sampling media.

1. Polyethylene bottles for inorganic analytes
2. Amber 1-liter glass jars with Teflon lids for inorganic and organic analyses requiring lower volumes of sample during extraction or digestion
3. Ziplock bags for biota samples, which are first wrapped in foil that has been double rinsed with deionized (DI) water
4. Amber 40-ml glass vials with Teflon septa for volatile organic analyses
5. Amber 80-oz glass bottles with Teflon caps for high-volume extractable organic analyses

6. Amber 8-oz wide-mouth glass jars for organic and inorganic analyses of soil and solid media

5.1.2 Container Preparation

All sample containers must be cleaned according to PMRMA Chemical QA Plan procedures. If preservatives are necessary to maintain sample integrity, the appropriate preservatives will be added at the time of sample collection. Cleaning procedures are to be used on new bottles only, and reuse of bottles is expressly prohibited. Purchases of commercial bottles will be restricted to I-Chem 300- and 200-series bottles, which are certified as analyte free for all project target compounds. Use of any other commercially available sample bottles may be acceptable if approved by PMRMA. The accepted bottle cleaning procedure for bottles other than I-Chem 300- and 200-series bottles or the equivalent is outlined below:

- Polyethylene bottles and polyethylene caps
  - Rinse bottles and lids with 5 percent sodium hydroxide
  - Rinse with DI water
  - Rinse with 5 percent Ultrex (or equivalent) nitric acid in DI water
  - Rinse with DI water
  - Drain and air dry
- Amber glass bottles or 40-ml vials
  - Scrub and wash bottles in detergent
  - Rinse with copious amounts of distilled water
  - Rinse with acetone
  - Rinse with methylene chloride (Nanograde or equivalent)
  - Rinse with hexane (Nanograde or equivalent)
  - Air dry
  - Heat to 200°C
  - Allow to cool
  - Cap with clean Teflon-lined caps

- Bottle caps
  - Remove paper liners from caps
  - Wash with detergent
  - Rinse with distilled water
  - Dry at 40°C
- Teflon liners (avoid contact with fingers)
  - Wash with detergent
  - Rinse with distilled water
  - Rinse with acetone
  - Rinse with hexane (Nanograde or equivalent)
  - Air dry
  - Place liners in cleaned caps
  - Heat to 40°C for 2 hours
  - Allow to cool
  - Use to cap cleaned bottles

5.1.3 Collection, Preservation, and Holding Times for Volatile Organic Compounds in Solid and Aqueous Media

When sampling aqueous media for volatile constituents, it is critical that the sample be agitated as little as is possible and that no head space be left in the sample container during shipment and storage prior to analyses. Sample containers should be triple rinsed with the sample media when possible. Volatile samples should never be allowed to freeze and should be stored at 4°C and analyzed as soon as possible. Volatile aqueous samples are never filtered. If separate aqueous phases are present in a sample media, individual phased samples should be collected and analyzed. Tap water samples will be collected after water has been allowed to flow for at least 3 minutes. Vials will be filled allowing for no head space and as little agitation as possible. Sodium thiosulfate will be added to compensate for the presence of residual chlorine.

Soil and sediment sample handling should be minimized. Containers should be filled completely, and care should be taken to assure the airtight integrity of the sample vial seal.

Holding times are seven days for all aqueous media analyzed for volatile aromatics unless preserved with hydrochloric acid (HCL) or sodium thiosulfate. If preserved, samples may be analyzed up to 14 days after the date of collection. Solid media must be analyzed 14 days after sampling, and the addition of preservatives is not required. Samples will not be analyzed if holding times have expired, unless otherwise authorized by PMRMA.

5.1.4 Collection, Preservation, and Holding Times for Inorganic Parameters and Semivolatiles/ Pesticides in Solid, Aqueous, and Biota Sampling Media

Aqueous Samples

Sampling equipment such as bailers, pumps, tapes, ropes, and spoons, must never be solvent-rinsed or scrubbed with detergents at the sampling site. Equipment dedicated to the sample site need not be rinsed. Non-dedicated sampling equipment should be rinsed with distilled water or scrubbed if carry-over is suspected. Ground water or newly installed monitoring wells should be allowed at least two weeks of equilibration time prior to sampling. When sampling is initiated, five volumes of water must be purged from a well prior to sample collection. Temperature, conductivity, and pH measurements should then be obtained and logged on the appropriate sampling form. If a well goes dry during pumping or bailing, it should be allowed to recover to its original water level. The investigative sample should be collected, followed by collection of a field sample for temperature, conductivity, and pH. Investigative samples should be filtered as appropriate (see Appendix C). Inorganic samples will usually be filtered prior to the introduction of preserving agents. Samples collected for metals analysis by inductively coupled plasma (ICP) emission spectrometer will be preserved in the field to a pH less than 2 using nitric acid. Holding times for ICP metals is six months. Anions or general water-chemistry samples will not be chemically preserved. Other specialized inorganic analytes will be preserved, and holding times will be observed as indicated in Appendix C. Semivolatile and pesticide samples will be collected in amber glass vials with Teflon-lined caps. No additives will

be used, but samples will be stored at 4°C and must be shielded from light until they are analyzed. Extraction holding times are 7 days from the date of sampling for semivolatiles and pesticides. Surface-water samples will require rinsing of sample containers downstream of the sampling location, and sampling equipment must be shielded from windblown sources of contamination. Tap-water samples will be collected after allowing water to run for 2 to 3 minutes. Sampling vials will be triple rinsed with the sampling media and filled completely. Rinsing should preclude filtering or preservation of the sampling media.

Solids

Soil and sediment samples must be collected such that sample integrity is not compromised prior to analysis. The Site Supervisor should instruct all field personnel as to the analytes of interest and proper use of the sampling apparatus. Prior to sampling, surface soil, vegetation, rocks, pebbles, leaves, twigs, and debris must be cleared from the site to allow for collection of a representative soil sample. Characteristic features such as staining, soil color, grain size, and sorting should be entered into a field logbook. Sampling equipment must be rinsed with distilled or PMRMA-approved water away from the sampling site at a decontamination area.

Soil samples will be collected with a non-plastic split or solid-core barrel sampler. Split core barrels will be split and bottled immediately after reaching the surface. Stainless-steel core barrels will be sealed immediately with Teflon-lined caps and taped shut with electrical tape. Sediment samples from a pond or lagoon should be rinsed with water from the surrounding environment; however, care should be taken to not disturb sediments near the sampling site.

Holding times for semivolatile and pesticide solid samples are 7 days. Generally chemical preservation is not required for soils and sediments samples. Refrigeration in a container that shields samples from light at 4° C until analysis is completed is an adequate method of preservation.

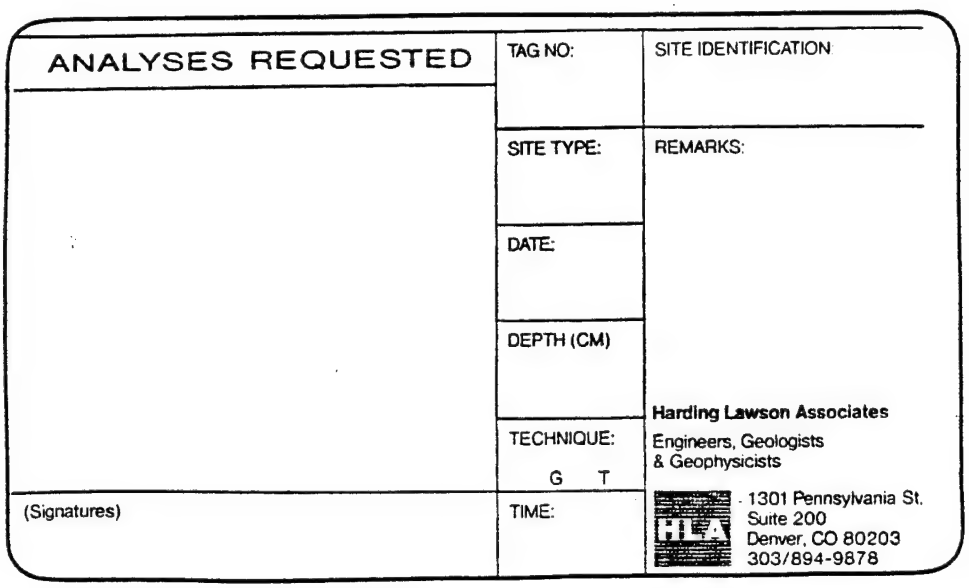
Biota

Biota samples will be trapped live, transported to a biopsy laboratory for dissection, and transported to an analytical laboratory for testing. If samples are found dead, they will be wrapped in double rinsed aluminum foil, placed in a plastic bag, and frozen until digested for analysis. Holding times for biota are four years.

5.2 SAMPLE MANAGEMENT

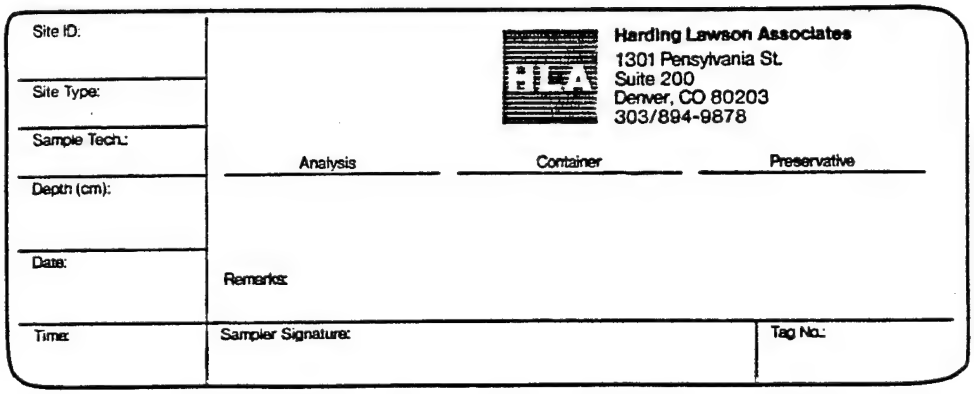
Samples collected in the field will be labeled and tracked according to matrix. Water samples will be tracked separately from soil, sediment, and biota samples. Sample labels and chain-of-custody (COC) forms will contain the same type of information for all media. Figures 5.1 through 5.4 provide examples of sample labels and COC forms that will be used in sampling each of these media. Each sample label and COC form will contain the following information:

1. Sample site type, which defines the nature of the media sampled (COC only)
2. Site ID, which is keyed into a location with a five-character alphanumeric code or by a descriptive title
3. Julian sampling date
4. Time of sample collection, in 24-hour clock designation
5. Depth from which the sample originated, if applicable
6. Sampling technique
7. Name and signature of personnel responsible for sample collection
8. Specific name of the chemical or physical analysis to be performed
9. Project name (COC only)
10. Airbill number (COC only)
11. Laboratory I.D. (COC only)
12. Sample label or tag number
13. Preservation technique used (filtered/unfiltered)



Prepared for:
Program Manager for
Rocky Mountain Arsenal
Commerce City, Colorado

Figure 5.1
SOILS/SOLIDS SAMPLE LABEL



Prepared for:
Program Manager for
Rocky Mountain Arsenal
Commerce City, Colorado

Figure 5.2
WATERS/LIQUIDS SAMPLE LABEL

Harding Lawson Associates
1301 Pennsylvania St
Suite 200
Denver, Colorado 80203
303/894-9878
Telecopy 303/894-9948

CHAIN OF CUSTODY RECORD

Lab: _____

Project No.	Project Name:	Sample Date:

Samplers: *(Signature)*

SITE TYPE	SITE IDENTIFICATION	TIME *(Military Standard)*	SAMPLE DEPTH *(Centimeters)*	SAMPLE TECHNIQUE	ANIONS	VOLATILE AROMATICS	ARSENIC	HYDROCARBONS	ORGANOCHLORIN PEST.	DBCP	PHOSPHONATES	VOLATILE HALOGENS	HYDRAZINE	MERCURY	ICP METALS	NITROSAMINES	NITRATES	ON/OP PESTICIDE	ORGANOACID	ORGANO SULFUR PEST.	SEMI-VOLATILES	THIODIGLYCOL	VOLATILES	NUMBER OF CONTAINERS	SAMPLE TAG NUMBER	REMARKS

ANALYSIS REQUIRED

Relinquished by: *(Signature)*	Date/Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date/Time	Received by: *(Signature)*
Relinquished by: *(Signature)*	Date/Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date/Time	Received by: *(Signature)*
Relinquished by: *(Signature)*	Date/Time	Received by: *(Signature)*	Date/Time	SAMPLE SPLIT DATE _____	
Relinquished by: *(Signature)*	Date/Time	Received by: *(Signature)*	Date/Time	SAMPLE EXTRACTION DATE _____	
				SAMPLE ANALYSIS DATE _____	
				AIR BILL NUMBER _____	

Laboratory Copy White — Project Office Copy Yellow — Field or Office Copy Pink

2001H

30C

Prepared for:
Program Manager for
Rocky Mountain Arsenal
Commerce City, Colorado

Figure 5.3
SOILS/SOLIDS CHAIN OF CUSTODY FORM

Harding Lawson Associates
1301 Pennsylvania St.
Suite 200
Denver, Colorado 80203
303/894-9878
Telecopy 303/894-9948

CHAIN-OF-CUSTODY RECORD

Lab ID:	Project Name:	Sample Date:	Site Type:	Site Identification:

Samplers: (Signature)	Sample Depth: (cm)	Sample Technique:

TIME	TAG NO.	ANALYSIS REQUIRED	CONTAINER	PRESERVATIVE/REMARKS

Relenquished by: (Signature)	Date/Time	Received by: (Signature)
Relenquished by: (Signature)	Date/Time	Received by: (Signature)
Relenquished by: (Signature)	Date/Time	Received by: (Signature)
Relenquished by: (Signature)	Date/Time	Received by: (Signature)

Airbill Number

Laboratory Copy White | Project Office Copy Yellow | Field or Office Copy Pink

3509 H

Prepared for:
Program Manager for
Rocky Mountain Arsenal

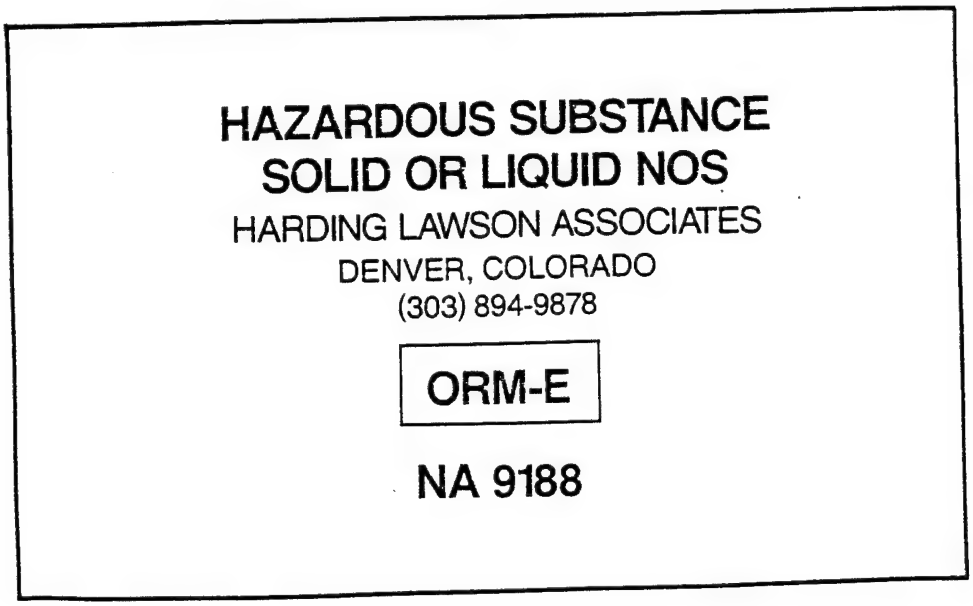
Commerce City, Colorado

Figure 5.4
WATERS/LIQUIDS CHAIN OF CUSTODY FORM

The sample label will be placed on the sample container prior to sample collection, when possible. The label will be secured to the container with waterproof tape, and information will be written in permanent ink prior to sample collection. Upon depositing the sample into the sample container, evidence tape will be attached to the sample container lid to assure the integrity of the sample during transport to the laboratory.

From the time of collection to final disposal of the samples, sample custody will be tracked on the HLA data management system (DMS). Information provided on the COC forms will be entered into the HLA DMS in the field, as described in the RIFS1 Data Management Plan. Samples will remain in the Site Supervisor's custody or control prior to being released or dispatched. The Site Supervisor will inspect each COC form to assure that it is accurate and legible prior to being released for transport to the laboratory. A hazardous materials label will be placed on a readily visible portion of the cooler to warn transportation personnel of its contents (Figure 5.5). The courier will obtain custody of the samples through an airbill similar to the one shown in Figure 5.6. A copy of this bill and a copy of the field COC form will be supplied to the QAC. Individuals relinquishing custody and receiving custody will sign, date, and record the time on the COC form or airbill, if applicable. Copies of the COC form and airbill will be placed inside the cooler in a tamper-proof envelope taped in place.

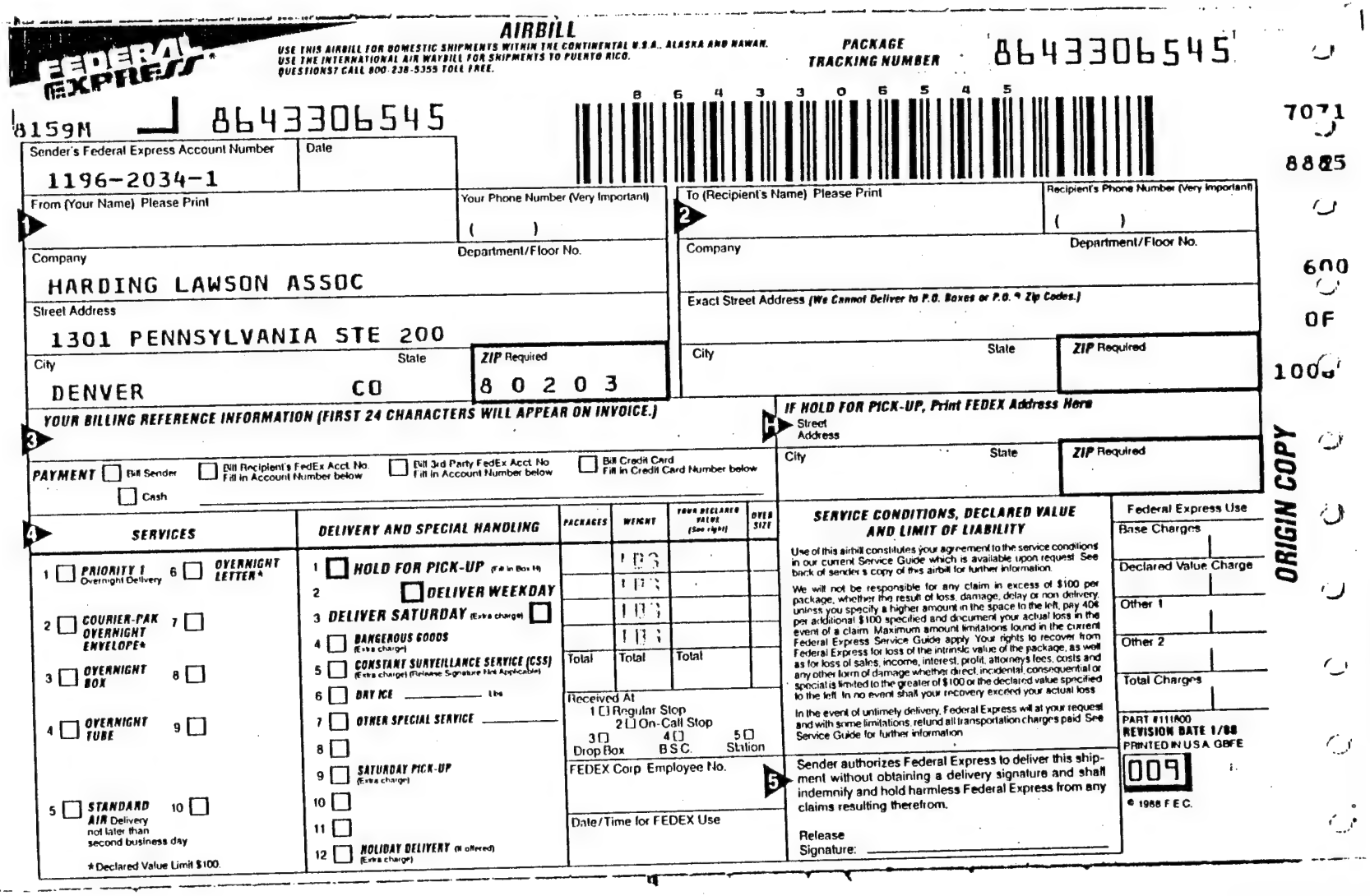
Upon arrival at the laboratory, sample cooler seals will be broken by the laboratory sample custodian, and COC forms and sample containers will be inspected. COC forms will be inspected for breakage, consistency, and legibility, and sample containers will be inspected for integrity before signing the airbill and COC form. Any inconsistencies or sample breakage will be reported immediately to the QAC. A sample lot designation and internal chain-of-custody (electronic or manual) procedure will be used by the laboratory to track investigative samples in the laboratory. Internal laboratory tracking standard operating procedures (SOPs) or lot designation forms will be provided to the QAC on a periodic basis to track analytical progress.



Prepared for:
Program Manager for
Rocky Mountain Arsenal
Commerce City, Colorado

Figure 5.5
WARNING LABEL FOR SAMPLE SHIPMENT

20000,140.10



FEDERAL EXPRESS

AIRBILL

USE THIS AIRBILL FOR DOMESTIC SHIPMENTS WITHIN THE CONTINENTAL U.S.A., ALASKA AND HAWAII.
USE THE INTERNATIONAL AIR WAYBILL FOR SHIPMENTS TO PUERTO RICO.
QUESTIONS? CALL 800-238-5355 TOLL FREE.

PACKAGE TRACKING NUMBER 8643306545

8159M 8643306545

8 6 4 3 3 0 6 5 4 5

7071 8825 600 OF 1000

Sender's Federal Express Account Number: 1196-2034-1

Date:

From (Your Name) Please Print:

Your Phone Number (Very Important): ( )

Company: HARDING LAWSON ASSOC

Department/Floor No.:

Street Address: 1301 PENNSYLVANIA STE 200

City: DENVER State: CO ZIP Required: 8 0 2 0 3

To (Recipient's Name) Please Print:

Recipient's Phone Number (Very Important): ( )

Company:

Department/Floor No.:

Exact Street Address (We Cannot Deliver to P.O. Boxes or P.O. + Zip Codes.):

City: State: ZIP Required:

YOUR BILLING REFERENCE INFORMATION (FIRST 24 CHARACTERS WILL APPEAR ON INVOICE.)

IF HOLD FOR PICK-UP, Print FEDEX Address Here

Street Address:

City: State: ZIP Required:

PAYMENT ☐ Bill Sender ☐ Bill Recipient's FedEx Acct. No. Fill in Account Number below ☐ Bill 3rd Party FedEx Acct. No. Fill in Account Number below ☐ Bill Credit Card Fill in Credit Card Number below ☐ Cash

SERVICES	DELIVERY AND SPECIAL HANDLING	PACKAGES	WEIGHT	YOUR DECLARED VALUE (See right)	OVER SIZE
1 ☐ PRIORITY 1 Overnight Delivery	1 ☐ HOLD FOR PICK-UP (Fill in Box H)		LBS		
2 ☐ COURIER-PAK OVERNIGHT ENVELOPE*	2 ☐ DELIVER WEEKDAY		LBS		
3 ☐ OVERNIGHT BOX	3 ☐ DELIVER SATURDAY (Extra charge)		LBS		
4 ☐ OVERNIGHT TUBE	4 ☐ DANGEROUS GOODS (Extra charge)		LBS		
5 ☐ STANDARD AIR Delivery not later than second business day	5 ☐ CONSTANT SURVEILLANCE SERVICE (CSS) (Extra charge) (Release Signature Not Applicable)	Total	Total	Total	
6 ☐ LETTER*	6 ☐ DRY ICE ____ Lbs				
7 ☐	7 ☐ OTHER SPECIAL SERVICE				
8 ☐	8 ☐				
9 ☐	9 ☐ SATURDAY PICK-UP (Extra charge)				
10 ☐	10 ☐				
	11 ☐				
	12 ☐ HOLIDAY DELIVERY (If offered) (Extra charge)				

*Declared Value Limit $100.

Received At: 1 ☐ Regular Stop 2 ☐ On-Call Stop 3 ☐ Drop Box 4 ☐ B.S.C. 5 ☐ Station

FEDEX Corp Employee No.:

Date/Time for FEDEX Use:

SERVICE CONDITIONS, DECLARED VALUE AND LIMIT OF LIABILITY

Use of this airbill constitutes your agreement to the service conditions in our current Service Guide which is available upon request. See back of sender's copy of this airbill for further information.

We will not be responsible for any claim in excess of $100 per package, whether the result of loss, damage, delay or non delivery, unless you specify a higher amount in the space to the left, pay 40¢ per additional $100 specified and document your actual loss in the event of a claim. Maximum amount limitations found in the current Federal Express Service Guide apply. Your rights to recover from Federal Express for loss of the intrinsic value of the package, as well as for loss of sales, income, interest, profit, attorney's fees, costs and any other form of damage whether direct, incidental, consequential or special is limited to the greater of $100 or the declared value specified to the left. In no event shall your recovery exceed your actual loss.

In the event of untimely delivery, Federal Express will at your request and with some limitations, refund all transportation charges paid. See Service Guide for further information.

5 Sender authorizes Federal Express to deliver this shipment without obtaining a delivery signature and shall indemnify and hold harmless Federal Express from any claims resulting therefrom.

Release Signature: _____

Federal Express Use

Base Charges

Declared Value Charge

Other 1

Other 2

Total Charges

PART #111800
REVISION DATE 1/88
PRINTED IN U.S.A. GBFE

009

© 1988 F.E.C.

ORIGIN COPY

U U 8643306545 8643306545

31B

Prepared for:
Program Manager for
Rocky Mountain Arsenal

Commerce City, Colorado

Figure 5.6
SAMPLE COURIER AIR BILL FORM

20000.140.10

Upon completion of analysis and final elevation of analytical results to a usable or non-usable status, samples will be released by the laboratory for transport back to RMA for storage and ultimate disposal. At this point, HLA will terminate the sample tracking processes.

6.0 ANALYSES

Analyses performed during offpost investigations will be performed using only Class 1, Class 1B, and Class 1A certified methods. Media to be analyzed include water, soil/sediment, and biota. The proposed project target analyte list for each environmental medium is shown by media in Figure 6.1. Methods will be selected such that the lowest achievable CRL for each media is realized. Methods will be selected on the basis of media and target analytes selected during each phase of the investigations. A general list of target analytes and probable associated methods of analysis are also shown in Table 6.1.

Aqueous and solid samples will be analyzed within analyte-specifc holding times described in Appendix C. Holding time limitations are defined as the maximum allowable time a sample may be stored for a given method of preservation prior to extraction or analysis. Samples analyzed after the specified holding time has been expired will be considered out-of-control, and results will be deemed unacceptable for inclusion into the RMA data base.

Biota holding times, shown in Appendix C, are four years. Samples will be wrapped in foil, when applicable, for immediate digestion. Digested biota samples will be stored frozen until extracted.

Standard solutions or investigative samples requiring dilutions will be prepared for organic analyses with ASTM Type II grade water. For inorganic standards and sample dilutions, ASTM Type I grade water will be used.

Aqueous samples to be analyzed for inorganics will be filtered in the field prior to the addition of nitric acid or other preservatives. Samples to be analyzed for organics will under no circumstances be filtered in the field. Inorganic samples to be analyzed for dissolved metals will be filtered in the field using a 0.45-micron fiber filter. Silicon fiber or cellulose acetate filters will be used to filter all samples except those designated for organic analysis. Specific types of water samples to be analyzed for organics other than oil and grease or volatiles will be filtered in the laboratory using a compatible filter. Filter material is considered compatible if the filter

Table 6.1: Target Analyte List

	Ground Water/ Tap Water	Surface Water	Soil/Stream and Pond Sediment	Surface Sediment	Biota	Method*
Volatile Organic Compounds						
1,1-Dichloroethane	x	x	x	-	-	GCCON/GCMS
1,2-Dichloroethane	x	x	x	-	-	GCCON/GCMS
1,1-Dichloroethene	x	x	x	-	-	GCCON/GCMS
1,1,1-Trichloroethane	x	x	x	-	-	GCCON/GCMS
1,1,2-Trichloroethane	x	x	x	-	-	GCCON/GCMS
Benzene	x	x	x	-	-	GCPID/GCMS
Bicycloheptadiene	x	x	x	-	-	GCPID/GCMS
Carbon Tetrachloride	x	x	x	-	-	GCCON/GCMS
Chlorobenzene	x	x	x	-	-	GCCON/GCMS
Chloroform	x	x	x	-	-	GCCON/GCMS
Dibromochloropropane	x	x	x	-	-	GCEC/GCMS
Dicyclopentadiene	x	x	x	-	-	GCFID/GCMS
Dimethyldisulfide	x	x	x	-	-	GCFPD/GCMS
Ethylbenzene	x	x	x	-	-	GCPID/GCMS
m-Xylene	x	x	x	-	-	GCPID/GCMS
Methylene chloride	x	x	x	-	-	GCCON/GCMS
Methylisobuytl ketone	x	x	x	-	-	GCFID/GCMS
o,p-Xylene	x	x	x	-	-	GCPID/GCMS
Tetrachloroethene	x	x	x	-	-	GCCON/GCMS
Toluene	x	x	x	-	-	GCPID/GCMS
1,2-Dichloroethene	x	x	x	-	-	GCCON/GCMS
Trichloroethene	x	x	x	-	-	GCCON/GCMS
Vinyl Chloride	x	x	x	-	-	GCEC/GCMS
Semivolatile Organic Compounds/Pesticides						
1,4-Oxathiane	x	x	x	x	-	GCFPD/GCMS
p-p'-DDE	x	x	x	x	x	GCEC/GCMS
p-p'-DDT	x	x	x	x	x	GCEC/GCMS
Aldrin	x	x	x	x	x	GCEC/GCMS
Atrazine	x	x	x	x	-	GCNPD/GCMS
Benzothiazole	x	x	x	x	-	GCFPD/GCMS
Chlordane	x	x	x	x	x	GCEC/GCMS
Chlorophenylmethyl sulfide	x	x	x	x	-	GCFPD/GCMS
Chlorophenylmethyl sulfoxide	x	x	x	x	-	GCFPD/GCMS
Chlorophenylmethyl sulfone	x	x	x	x	-	GCFPD/GCMS
Dieldrin	x	x	x	x	x	GCEC/GCMS
Diisoproylmethylphosphonate	x	x	x	x	-	GCFPD/GCMS

Table 6.1: (Continued)

	Ground Water/ Tap Water	Surface Water	Soil/Stream and Pond Sediment	Windblown Sediment	Biota	Method*
Semivolatile Organic Compounds/Pesticides (continued)						
Dimethylmethylphosphonate	x	x	x	x	-	GCFPD/GCMS
Dithiane	x	x	x	x	-	GCFPD/GCMS
Endrin	x	x	x	x	x	GCEC/GCMS
Hexachlorocyclopentadiene	x	x	x	x	x	GCEC/GCMS
Isodrin	x	x	x	x	x	GCEC/GCMS
Malathion	x	x	x	x	-	GCNPD/GCMS
Parathion	x	x	x	x	-	GCNPD/GCMS
Supona	x	x	x	x	-	GCNPD/GCMS
Vapona	x	x	x	x	-	GCNPD/GCMS
Phenol	x	x	x	-	-	GCFID/GCMS
2-Chlorophenol	x	x	x	-	-	GCFID/GCMS
2-Nitrophenol	x	x	x	-	-	GCFID/GCMS
2,4-Dimethylphenol	x	x	x	-	-	GCFID/GCMS
2,4-Dichlorophenol	x	x	x	-	-	GCFID/GCMS
4-Chloro-3-methylphenol	x	x	x	-	-	GCFID/GCMS
2,4,6-Trichlorophenol	x	x	x	-	-	GCFID/GCMS
2,4-Dinitrophenol	x	x	x	-	-	GCFID/GCMS
4-Nitrophenol	x	x	x	-	-	GCFID/GCMS
4,6-Dinitro-2-methylphenol	x	x	x	-	-	GCFID/GCMS
Pentachlorophenol	x	x	x	-	-	GCFID/GCMS
Inorganics/General Characteristics						
Cadmium	x	x	x	x	-	ICP
Calcium	x	x	-	-	-	ICP
Chromium	x	x	x	x	-	ICP
Copper	x	x	x	x	-	ICP
Sodium	x	x	-	-	-	ICP
Lead	x	x	x	x	-	ICP
Magnesium	x	x	-	-	-	ICP
Potassium	x	x	-	-	-	ICP
Zinc	x	x	x	x	-	ICP
Arsenic/AA	x	x	x	x	x	EPA-206
Mercury/AA	x	x	x	x	x	EPA-245
Total amenable cyanide	x	x	-	-	-	EPA-335.1
Biological Oxygen Demand	-	x	-	-	-	EPA-405.1

Table 6.1: (Continued)

	Ground Water/ Tap Water	Surface Water	Soil/Stream and Pond Sediment	Windblown Sediment	Biota	Method*
Inorganics/General Characteristics (continued)						
Sulfide	-	x	-	-	-	EPA-376.2
Ammonia Nitrogen	-	x	x	-	-	EPA-350.1
TOC	x	x	x	-	-	EPA-415.2
Alkalinity	x	x	-	-	-	Alkalinity
Nitrate/Nitrite	x	x	-	-	-	EPA-300
Sulfate	x	x	-	-	-	EPA-300
Chloride	x	x	-	-	-	EPA-300
Fluoride	x	x	-	-	-	EPA-300
pH	x	x	-	-	-	EPA-150.1
Specific Conductance	x	x	-	-	-	EPA-120.1
Temperature	x	x	-	-	-	EPA-170.1
Dissolved Oxygen	-	x	-	-	-	EPA-360.2

Abbreviated Method Name Descriptions:

AA - Atomic Absorption Spectroscopy

GCCON - Gas Chromatography/Conductivity Detector

GCEC - Gas Chromatography/Electron Capture

GCFID - Gas Chromatography/Flame Ionization Detector

GCFPD - Gas Chromatography/Flame Photometric Detector

GCMS - Gas Chromatography/Mass Spectrometry

GCNPD - Gas Chromatography/Nitrogen Phosphorous Detector

GCPID - Gas Chromatography/Photoionization Detector

ICAP - Inductively Coupled Plasma Emission Spectrometry

* Methods to be employed for the analyses of program sampling media will be determined using the following criteria: (1) desired certified reporting limit (CRL) to meet program and PMRMA requirements; (2) effectiveness of the method in analyzing a sampling media; (3) availability of a certified method to analyze a sample media; and (4) need to confirm GC results using GC/MS

material is not changed by the material being filtered and the filter material does not leach or adsorb the analytes of interest. The method blank must also be filtered in the same manner as the investigative samples to isolate any carry-over or constituent introduction that may be attributable to the filtration process.

Solid or soil samples display a wide range of physical characteristics. Soil samples will be described carefully in the field for such physical characteristics as grain size, mineralogy, sorting, color, odor, and organic content. Windblown sediments and surface grab samples will be collected when possible from surfaces that are relatively free of surface vegetation. Sludge or boggy soils will be sampled in a manner that minimizes free water collection, but matrix-bound water will not be removed. Soils will be analyzed in their native condition except that nonvolatile organic samples will be homogenized in the laboratory by stirring or shaking prior to splitting the sample for analysis. A split of each reserved soil sample will be dried using ASTM procedure D2216-71 to estimate the moisture content. The percent moisture content will be calculated using the following equation, and the result will be entered into the PMRMA IRDMS. Weighed aliquot fractions of the mixed sample will be analyzed and reported in the condition received.

$$\frac{\text{Sample Wet Weight - Sample Dry Weight}}{\text{Sample Wet Weight}} \times 100$$

Aqueous, solid, and biota samples will be split into separate analytical lots because methods used in extraction and processing differ by sample medium. Biota samples will also be treated as separate lots and will be analyzed independent of other media.

Analytical lot designations for each medium type are defined as the total number of samples that can be processed by a certified method in a 24-hour period, including all relevant QC samples. Limiting factors may dictate smaller lots than can be physically processed by a method. A lot size cannot be increased unless a new method is developed to increase overall method productivity or an alternate certified method can be employed.

All instrument measurements must be within the certified reporting range of a method. Samples requiring dilution must be flagged, and dilution factors must be recorded and entered into the PMRMA IRDMS. If a large number of investigative samples require dilution, the PMRMA Project QA Officer will decide whether the method certification range should be extended. Samples that cannot be run with the required holding time and within the method certified reporting range should not be analyzed. In such cases, the QAC should be informed immediately so that the appropriate corrective action can be implemented.

6.1 CALIBRATION CURVES

Prior to sample analysis, calibration standards of each target analyte must be analyzed to establish that the instrument is functioning properly with the desired sensitivity. To determine this, two additional types of calibrations will be required outside of precertification and certification calibration procedures. These additional calibrations include a multilevel initial calibration and a single concentration level daily continuing calibration. Calibration standards will be prepared independently from stock solutions, other than those used to prepare certification calibration standards. Initial calibration concentrations will be selected such that they bracket the certified range of the method. Initial calibration data need not be plotted, but the response versus concentration relationship should be used to provide an early warning of excessive instrument variance. Instrument calibration curves and data will not to be used in calculating method certified reporting limits (CRLs). For all classes of methods except Class 2 methods, two-thirds of the analytes must meet the required calibration response factor percentage requirements (relative response factor >10 percent of mean response factor for inorganics and >25 percent for organics) or be within two standard deviations for the method to be considered in control. Analytes that fail to meet this criteria must be re-analyzed.

6.1.1 Initial Calibration Class 1, Class 1A and Class 1B Methods

Initial calibrations procedures must be performed the first day that certification analyses are performed. These procedures must be repeated when one of the following situations arises:

1. The instrument is started up after replacement of parts or after a period of inactivity
2. A new group of analytes is to be analyzed
3. The instrument fails to meet the daily continuing calibration criteria

A minimum of one reagent blank and five calibration standards will be analyzed for Class 1 methods or one blank and three calibration standards for Class 1 and Class 1B methods at concentrations that bracket the method certified reporting range. In all cases, standards will be analyzed during a single day. Concentrations may be prepared directly in solvent as if they had been subjected to the entire series of method extraction and preparation steps. If the certified reporting range of a method has been extended by the addition of certification samples, the same number of additional calibration standards must also be included.

All standards will be prepared independently from a concentrated stock solution; duplicate instrumental analyses of a single set of standards are not acceptable. Table 6.2 shows lists total number and concentrations required for initial and continuing calibration procedures.

If samples are analyzed on the same day that the initial calibration is performed, one standard at the highest concentration must be analyzed after sample analyses are completed. For the first seven calibrations, the response must agree (within 10 percent for inorganic analyses in surface/ground water and within 25 percent for all other analyses) with the mean response for the same concentration, as determined from precertification and the certification initial calibration. After seven calibrations, responses must agree within two standard deviations rather than a certain percentage. If the response factors fail the test, the daily standard will be re-analyzed. If the response from the second analysis is not within the required percentage or two standard deviations of the mean response from precertification and the certification initial calibration, the system is considered to have failed calibration. All data generated since the last satisfactory calibration are considered questionable and must be re-analyzed after repeating the initial calibration.

Table 6.2: Numbers and Concentrations of Calibration Standards
(Linear and Zero-Intercept)

INITIAL CALIBRATION - CLASS 1

Minimum Testing Range (MTR); 7 Standards + 2 Check Standards (CS)
Blank, *0.5, 1, 2, 5, *10, & *10 TRL + CS & CS
MTR + 1 Order of Magnitude Extension: 10 Standards + 2 Check Standards
Blank, *0.5, 1, 2, 5, 10, 20, 50, *100, & *100 TRL + CS & CS
MTR + 2 Orders of Magnitude Extension: 13 Standards + 2 Check Standards
Blank, *0.5, 1, 2, 5, 10, 20, 50, 100, 200, 500, *1000, & *1000 TRL + CS & CS

INITIAL CALIBRATION - CLASS 1A

Minimum Testing Range (MTR); 5 Standards
Blank, *0.5, 2, *10, & *10 TRL
MTR + 1 Order of Magnitude Extension: 7 Standards
Blank, *0.5, 2, 10, 50, *200, & *200 TRL
MTR + 2 Orders of Magnitude Extension: 9 Standards
Blank, *0.5, 2, 10, 50, 200, 500, *2000, & *2000 TRL

INITIAL CALIBRATION - CLASS 1B

Minimum Testing Range (MTR); 5 Standards + 1 Check Standard (CS)
Blank, *0.5, 2, *10, & *10 TRL + CS
MTR + 1 Order of Magnitude Extension: 7 Standards + 1 Check Standard
Blank, *0.5, 2, 10, 50, *200, & *200 TRL + CS
MTR + 2 Orders of Magnitude Extension: 9 Standards + 1 Check Standard
Blank, *0.5, 2, 10, 50, 200, 500, *2000, & *2000 TRL + CS

INITIAL CALIBRATION - CLASS 2
Minimum Testing Range: 6 Standards
Blank and 1 TRL (Triplicate)

DAILY CALIBRATION - CLASS 1/CLASS 1A/CLASS 1B

Minimum Testing Range (MTR); 2 Standards
*10 & *10 TRL
MTR + 1 Order of Magnitude Extension: 2 Standards
*100 & *100 TRL
MTR + 2 Orders of Magnitude Extension: 2 Standards
*1000 & *1000 TRL

DAILY CALIBRATION - CLASS 2
Minimum Testing Range
Blank and 1 TRL (Duplicate)

*10% to 25% Range Extension

For Class 1 and 1B, method calibration check standards must also be analyzed with the initial calibration standards. Calibration check standards can be obtained from EPA or other commercial sources and should contain target analytes at concentrations near the high end of the calibration range. For Class 1 analysis, two calibration check standard analyses are required, one at the beginning of each day and one at the end of each day. In Class 1B analyses, a calibration check standard is required only at the beginning of each day. Calibration check standards must meet the manufacturer's acceptance criteria. If the check sample(s) do not meet these criteria, the sample(s) may be re-analyzed once. Should the re-analysis fail to satisfy the specified criteria, the initial calibration is considered unacceptable and corrective action must be initiated.

6.1.2 Daily Calibration, Class 1, Class 1A, Class 1B, and Class 2

Calibration standards will be analyzed each day to verify that instrument response has not changed from the previous calibration. The highest concentration standard will be analyzed, and results must fall within the specified percentage or two standard deviation criteria for all previous calibrations and certification results. If, after a single re-analysis is performed, the standard does not meet these criteria, a new initial calibration must be performed. After investigative sample analyses are completed each day, the highest concentration standard must be similarly analyzed and meet performance criteria. If the standard does not meet these criteria after the second analysis is complete, the calibration is considered invalid and all related investigative samples must be re-analyzed under a new calibration. The above procedures apply only to linear or zero-intercept calibration curves. For non-linear and non-zero-intercept calibrations, a low, middle, and high calibration standard must be analyzed and be within two standard deviations of all previous calibrations and certification results. Calibrations fitted by a quadratic equation require a minimum of four standards over the certified range, with the highest concentration analyzed at the end of each day. Class 2 methods must be calibrated in triplicate, and blanks must yield negative results during the initial calibration. Before and after each analysis day, a calibration standard at a concentration equal to the method CRL and a blank must be analyzed. If the blank

contains positive results and the calibration standard fails to meet the standard deviation criteria, a single re-analysis is permitted. Should any of these samples fail to meet the specified criteria during re-analysis, a new initial calibration must be performed.

6.2 REFERENCE MATERIALS

Three types of reference materials may be used in preparing certification samples, calibration standards, and investigative samples for analyses. These types of material include Standard Analytical Reference Materials (SARMs), Interim Reference Materials (IRMs), and off-the-shelf materials. Whenever possible, reference materials used to prepare SARMs should be supplied by the USATHAMA Central QA Laboratory. These reference materials are developed from National Bureau of Standards (NBS) standard reference material analyses that have been run a sufficient number of times to document random as well as systematic errors in results. Each of these standards is also subjected to a storage period to estimate SARM stability. The USATHAMA Central QA Laboratory will also store SARMs and inform the contractors when SARMs have deteriorated below the 98 required mole percent purity.

When SARMs are not available, IRMs should be obtained for use from the Central QA Laboratory. IRMs are prepared by USATHAMA but have not been as rigorously evaluated or controlled as SARMs. Off-the-shelf materials should be used only when SARMs or IRMs are not available from USATHAMA. In this case, the contractor laboratory must analyze the material for purity and absence of target analytes. When these types of standard materials are used for method certification, purity characterization information must be provided with the certification performance data package. The contractor must also supply USATHAMA with 10 percent by weight of the raw materials used in preparing off-the-shelf IRMs.

7.0 SYSTEM CONTROLS

System controls are designed to monitor and document performance of the total measurement system. To comply with the PMRMA Chemical QA Plan, these controls are initiated throughout the collection and analyses of environmental samples. System controls are implemented to manage sample tracking, documentation of activities, results, and analytical performance. Sample tracking describes procedures to be followed through sample collection, transfer, analyses, and ultimate sample disposal. Document control monitors the storage of field data and raw analytical data from field collection, through reporting of results by the laboratory, and during use of these results. Analytical performance monitoring is accomplished through the use of QC samples, which are analyzed to provide quantitative evidence that the entire method is performing comparably to established QC criteria.

7.1 SAMPLE CONTROL

Investigative samples will be tracked with an automated sample tracking system developed by HLA for RIFS1. The system is capable of tracking sample documentation and deliverables from sample collection, transfer, analysis, and ultimate sample disposal. This system is described in detail in the RIFS1 Data Management Plan. Procedures utilized by subcontractor laboratories to manage sample tracking are integrated into the HLA tracking system to ensure that sample custody is maintained. Each contractor laboratory will submit sample tracking procedures and documentation of control procedures to PMRMA as independent evidence of in-place system controls. Field and laboratory audit procedures outlined in Section 9.0 of this document detail the methods that will be employed by HLA to assure that appropriate field and laboratory documentation is being preserved for final retrieval by PMRMA.

Sample control procedures to be utilized by HLA for field activities are presented in Section 5.0 of this document. Procedures to be used by each contractor laboratory must be consistent with standard chain-of-custody practices previously employed for RMA programs. Standard tracking procedures or SOPs will be provided by the subcontractor laboratories to HLA and must

be approved by the QAC and PMRMA prior to the analyses. Automated sample control programs may be substituted for manual chain-of-custody procedures but must provide the same information and high degree of reliability as a manual system. A laboratory tracking or sample control system must contain the following elements:

1. A Sample Receipt Officer or Custodian
2. An individual responsible for samples
3. Analyses request information
4. Field sample number
5. Location of sample storage at all times
6. Date sample was received or retrieved by analyst
7. Present condition or step of analysis being performed on a sample
8. Data analysis was completed
9. Signature of analysts
10. Laboratory ID number
11. Bottle tag number
12. Report date
13. Special instructions for analysis

Each laboratory must appoint a Laboratory Quality Assurance Coordinator (LQAC) to assure that the above information is collected and maintained throughout a project. This individual will be responsible for sample integrity while in the custody of the laboratory. The LQAC will maintain a permanent record of all identifying sample tags, data sheets, and laboratory records. Sample custodians and analysts will be responsible for samples in their custody. Sample custodians will be responsible for inspecting samples for breakage upon receipt at the laboratory. Any problems with project samples will be communicated by the LQAC to the QAC immediately after receipt and inspection of samples by the sample custodian. Samples will be logged into a separate RMA logbook that must contain the following information:

1. Unique laboratory sample ID number
2. RMA project name
3. Date and time of arrival
4. Analysis requested
5. Type and condition of sample container
6. Airbill number
7. Location where samples will be stored
8. Observations concerning sample condition, including broken containers, leakage, and temperature
9. Signature of sample custodian

The sample custodian will transfer custody of the samples to the appropriate section supervisor so that lot assignments and analysis request forms can be issued. Samples will be distributed into a secure storage facility, and the chain-of-custody forms will be signed by the appropriate section supervisor. Samples will be logged in or out of this storage facility for analyses or return to RMA. Any sample splitting or extraction will be logged onto the chain-of-custody form or laboratory extraction bench log. Extraction or processing logs will be stored with all other required raw data deliverables in a secure storage facility until the final data purge is performed by PMRMA. A more detailed description of recordkeeping requirements is provided is Section 9.0 of this QA Plan.

7.2 QUALITY CONTROL SAMPLES

Two types of QC samples will be employed to provide quantitative evidence of performance of field procedures and analytical methods. QC samples include external and laboratory samples that are introduced into the sample analysis stream. Laboratory QC samples are samples introduced into the sample train by the laboratory to monitor analytical performance. External QC samples are samples introduced into the sample train in the field to monitor both sample collection and analytical performance.

QC samples will be prepared in authentic blank media provided to each laboratory on a demand basis. The QAC will be responsible for providing the laboratory with sufficient materials to accomplish QC objectives. The purpose of QC samples is to monitor day-to-day variations in routine analyses. It is essential that controls are initiated during and maintained throughout analyses. The approach described in this QA Plan is intended to support, not replace, existing laboratory QC practices. Control samples will be prepared from standard matrices or actual field samples and will be processed through the entire method. The numbers and concentrations required for QC samples in each certification class are summarized by sample lot in Table 7.1. For Classes 1 and 1B, a method blank is required for each lot followed by the spiked QC samples, which contain all control analytes. The control analytes for each method are specified in the certification data package. For GC/MS Class 1A methods, a method blank spiked with the appropriate control compounds (surrogates only) will be run initially to evaluate system control and assure that the laboratory is not a source of contamination. For Class 2 methods, a method blank is required, followed by a standard control analyte spike at a concentration equal to the method CRL.

Sample numbers for QC samples will be assigned by the sample custodian during the log-in process. QC samples will be prepared by a standards preparation specialist or the analyst responsible for the first step of an analytical method. This individual will be responsible for obtaining the appropriate volume/weight and type of standard spike and matrix to be used. Spiking solutions and procedures must be identical to those submitted during method certification. All spiked QC samples, except those to be analyzed for volatiles or waters, must be allowed to equilibrate for one hour.

For Class 1 certified methods, the method blank will be followed by two QC samples run at a concentration near, but not above, the upper reporting limit (usually 10 times the CRL). The concentration of these two samples must be identical and will be reported to include the appropriate method-specified significant figures. A third spike will also be analyzed at a concentration near the project-specified action level, approximately two times the method CRL. Control charts

Table 7.1: Numbers and Concentrations of QC Samples per Lot

CLASS 1

1 - Standard Matrix Method Blank
3 - Standard Matrix Spikes
2, 10, & 10 CRL (approximate)

CLASS 1A

1 - Standard Matrix Method Blank/Spike
ALL - Natural Matrix (Field Sample) Spikes
10 CRL (approximate) Surrogate

CLASS 1B

1 - Standard Matrix Method Blank
1 - Standard Matrix Spike
10 CRL (approximate)

CLASS 2

1 - Standard Matrix Method Blank
1 - Standard Matrix Spike
1 CRL

will be prepared for each control analyte. A minimum number of in-control data values per lot can be viewed on these control charts and can be used to evaluate system stability. Several installations may be grouped per daily analytical lot; however, data entered into the IRDMS system must be sorted by installation.

Class 1A certified methods require that a method blank spiked with surrogates be analyzed per lot at a concentration near but not exceeding the upper reporting limit (usually 10 times the CRL). In addition, each investigative sample will be spiked with surrogate compounds at a concentration of 10 times the CRL or at the upper reporting limit (URL). As for all multilevel methods, the standard matrix-spiked sample recoveries will be plotted on control charts with minimum number of in-control points required for the method to be considered stable (see Table 7.2).

In Class 1B analyses, a separate QC sample containing all control analytes will accompany the method blank at a concentration of 10 times the URL. The method for plotting these analytes and evaluating them are similar to those described for method Classes 1 and 1A.

Class 2 methods require a method blank and a single control sample. No control charts can be generated for these method class analyses. Methods certified with extended reporting ranges must include one extra control sample at or near the CRL of the method. These values will also be plotted, and system control will be evaluated in the same manner as that for standard non-extended range methods.

Results for QC samples will not be corrected and no dilutions will be allowable. Analytical work will not proceed until each lot of QC samples has been reviewed and found to be acceptable within the limits for the analytical method.

Method blanks will be reported uncorrected, as determined on the basis of the instrument calibration response factor. Investigative samples will be corrected for blank artifacts detected above the CRL only. Blank artifacts detected at concentrations above the method CRL will be singularly subtracted from investigative sample results on a response level prior to determination of a found concentration. Investigative sample concentrations will then be calculated and entered

Table 7.2: Minimum Number of In-Control Points for Multi-Analyte Methods

Required Control Analytes Per Method	Required Number of Data Values Falling Between the UCL and LCL
1	1
2	2
3	2
4	3
5	4
6	4
7	5
8	6
9	6
10	7
11	8
12	8
13	9
14	10
15	10
16	11
17	12
18	12
19	13
20	14
21	14
22	15
23	16
24	16
25	17

as a found concentration into the IRDMS. Waters will be entered into the IRDMS in μg/l or ppb, and soil, sediments, and biota will be reported in μg/g or ppm. Blank contamination problems must be delineated by each laboratory. Unusual problems that arise during analyses must be discussed with the QAC. Allowable limits of blank contamination must be fully discussed and approved by the QAC prior to resuming work.

7.3 CONTROL CHARTS

Control charts are prepared only for Classes 1, 1A, and 1B certified methods. Control charts monitor and graphically display trends that may affect the precision or accuracy of routine analyses. Certification data are used to establish the control limits required for each QC control analyte. Data will be plotted onto control charts prior to corrections for accuracy. Control charts will consist of tabulated data and related graphical displays. Software programs for the production of control charts will be provided by PMRMA. Initial construction of control limits will be performed using statistical methods described in Section 4.0 of this QA Plan, and control limits will be applied to certification data packages. Initially, control limits derived for the certification data will be used to determine whether analytical systems are in control while investigative sample are being analyzed. The two basic types of control charts that will be used to perform this elevation are mean recovery (X-bar) control charts that plot the average or mean values of spike recoveries and/or range (R) control charts.

In both types of control charts, the range is a mean value plotted along the ordinate axis and the corresponding lot designations are plotted along the abscissa. X-bar control charts monitor method accuracy, and R control charts graphically portray method precision. Each control chart should contain the following required information:

1. Analyte
2. Method number
3. Laboratory
4. Spike concentration
5. Chart title

6. Three letter lot designation and analysis date for each point, shown on the x-axis
7. Percent recovery (for X-bar control charts) or Range (for R control charts) along the y-axis
8. Upper control limit (UCL) on X-bar and R control charts
9. Upper warning limit (UWL) on X-bar and R control charts
10. Mean on X-bar and R control charts
11. Lower warning limit (LWL) on X-bar control charts
12. Lower control limit (LCL) on X-bar control charts

Class 1 control charts must be produced for both the low and high spikes. For high spiked duplicated samples, X-bar is defined as X-double bar and is equivalent to the sum of percent recovery value divided by the total number of measurements performed. Percent concentration is defined by the following equation:

$$\text{Percent Recovery} = \frac{\text{Found Concentration - Method blank (Inst. Response)}}{\text{Spiked Concentration}} \times 100$$

For duplicate QC or multiple QC samples, range and average recoveries are defined by the following equation

$$\bar{\bar{X}} = \frac{\Sigma \bar{X}}{K} \quad \text{and} \quad \bar{R} = \frac{\Sigma R}{K}$$

where K is the cumulative number of QC example pairs in the data base. From statistical definitions described in Section 4.0, the control limits for paired results can be defined as follows:

Average Upper Warning limits = $UWL_{\bar{X}} = \bar{\bar{X}} + 1.25\,\bar{R}$

Average Upper Control limit = $UCL_{\bar{X}} = \bar{\bar{X}} + 1.88\,\bar{R}$

Average Lower Warning Limit = $LWL_{\bar{X}} = \bar{\bar{X}} - 1.25\bar{R}$

Average Lower Control limit = $LCL_{\bar{X}} = \bar{\bar{X}} - 1.88\,\bar{R}$

Upper Warning Limit on Range = $UWL_R = 2.511\bar{R}$

Upper Control Limit on Range = $UCL_R = 3.267\bar{R}$

By definition, there is no lower limit of range except zero with zero value corresponding with absolute correlation. In Appendix D and E, example control charts are provided for Class 1 and Class 1A certified methods. Control charts will be updated after each lot for the first 20 sample lots. Limits established after the first 20 lots will be used for the next 20 lot evaluations. Thereafter, control charts will be updated every 20 lots using the 40 most recent control points. New data must be combined with individual values for percent recovery, not mean or total previous data mean values. Only in-control lots are to be used in updating control limits. Outlier points should be plotted but not utilized in lot number requirements or control limit calculations. Outliers will be determined using Dixon's Test at the 98 percent confidence level. Dixon's Test expresses the gap between an outlier and the nearest value as a fraction of the range between the smallest and largest value. Average or range values are ordered with the highest values receiving a rank of 1 (x_1) and lowest values receiving a rank of N (x_n). Outliers (x_i or x_x) are then defined for a given number of analytical test results by the following equations. Critical values are designated as "r" for a given population. Critical values of r are provided in Table 7.3 for populations up to 25 test results.

For less than eight measurements, reject x_N if

$$\frac{x_N - x_{N-1}}{x_N - x_1} > r_{10} \quad \left(\text{or reject } x_1 \text{ if } \frac{x_2 - x_1}{x_N - x_1} > r_{10}\right)$$

Between eight and ten measurements, reject x_N if

$$\frac{x_N - x_{N-1}}{x_N - x_2} > r_{11} \quad \left(\text{or reject } x_1 \text{ if } \frac{x_2 - x_1}{x_{N-1} - x_1} > r_{11}\right)$$

Between 11 and 13 measurements, reject x_N if

$$\frac{x_N - x_{N-2}}{x_N - x_2} > r_{21} \quad \left(\text{or reject } x_1 \text{ if } \frac{x_3 - x_1}{x_{N-1} - x_1} > r_{21}\right)$$

Over 13 measurements, reject x_N if

$$\frac{x_N - x_{N-2}}{x_N - x_3} > r_{22} \quad \left(\text{or reject } x_1 \text{ if } \frac{x_3 - x_1}{x_{N-2} - x_1} > r_{22}\right)$$

Table 7.3: Critical Values for Dixon's Outlier Test
(a = 0.02)

Number of Measurements (N)	Criterion (r)	Critical Value of r
3	r_{10}	0.976
4		0.846
5		0.729
6		0.644
7		0.586
8	r_{11}	0.631
9		0.587
10		0.551
11	r_{21}	0.638
12		0.605
13		0.578
14	r_{22}	0.602
15		0.579
16		0.559
17		0.542
18		0.527
19		0.514
20		0.502
21		0.491
22		0.481
23		0.472
24		0.464
25		0.457

The critical values for the test statistic at 98 percent confidence level are also shown in Table 7.3. If the test statistic is greater than the critical value from the table, the data point is an outlier. Once adequate data are available, N will be kept constant at 20, with the 20 most recent data points. Once 60 or more lots have been analyzed, control limits must be recalculated on the 40 most recent lots. If a method is out of control, no point from the related analyses may be used to update the control charts.

Outliers are attributed to one of the following causes, which results in data that are unusable.

- A measurement that was incorrectly read, recorded, or transcribed
- A faulty instrument
- Incorrect calculations
- Incorrect application of an analytical method

The principal safeguards against obtaining or using an outlier are vigilance during all operations and visual inspection of data before performing statistical analyses.

Three-point moving average control charts will be employed when single QC spikes are being reviewed. This applies to Class 1 single low-concentration QC spikes, Class 1B single high-concentration spikes, Class 1A standard/blank QC samples, and additional extended range QC samples. Charts will be prepared similar to X-bar and R control charts except that subsequent points are obtained by averaging the three most recent individual recovery values (outliers included) and the range for each value is the difference between the highest and lowest value for each group of three values. Control and warning limits are also determined differently, using the same equations but adjusted statistical multipliers:

UWL on Average: $UWL_{\overline{X}} = \overline{\overline{X}} + 0.682\,\overline{R}$

UCL on Average: $UCL_{\overline{X}} = \overline{\overline{X}} + 1.023\,\overline{R}$

LWL on Average: $LWL_{\overline{X}} = \overline{\overline{X}} + 0.682\,\overline{R}$

LCL on Average: $LCL_{\overline{X}} = \overline{\overline{X}} + 1.023\,\overline{R}$

UWL on Range : $UWL_R = 2.050\,\overline{R}$

UCL on Range : $UCL_R = 2.050\,\overline{R}$

The Dixon Outlier Test will be applied to three-point moving averages, and outliers will be progressively eliminated until the three-point average is calculated from the three most recent in-control results.

Evaluations of out-of-control situations must be reviewed daily so that corrective action can be taken immediately. Failure to take immediate action may result in the need to discard large amounts of data and re-analyze samples. Out-of-control situations may include failure to meet calibration criteria, recordkeeping omissions, improper sampling technique, and improper storage or preservation of samples. Samples that have exceeded holding times are also out-of-control, and samples should not be processed unless written or verbal authorization is provided by the QAC. Control charts are also used to further refine out-of-control conditions. Average or X-bar control charts may indicate a pending problem, and the analyst or section supervisor should be contacted concerning the potential problems such as the following:

- A series of five successive points going in the same direction
- A cyclical pattern of control values
- Two consecutive points between the UWL and UCL or the LWL and LCL
- A value outside the control limits or classified as an outlier by statistical test
- A series of seven successive points on the same side of the central line

The analyst must terminate work if any of the following X-bar control chart conditions are recognized. These criteria will also be used by the QAC in reviewing control charts to determine and make a recommendation on the acceptability of analytical results.

1. Plot average percent recovery (X) for each analyte.
2. If the points for at least two-thirds (see Table 7.2) of the control analytes for a multi-analyte method are not classified as out-of-control, based on the conditions described above, the method is in control and environmental sample data may be reported (providing that condition 3 below has not occurred). The conditions that may have caused fewer than two-thirds of the control analytes to fail the control criteria will be investigated and corrected as necessary. All activities will be documented. The data points indicating possible error will be annotated with a reference to the investigation and to the fact that the method met control criteria.

3. A method may be deemed out of control even if greater than or equal to two-thirds of the control analytes meet control criteria. Of the remaining control analytes (less than one-third possible out-of-control), if one analyte has two consecutive out-of-control points, as defined above, the method is out of control. Analyses must cease, the cause must be investigated and corrected, and a determination must be made by the PMRMA Project QA Officer as to whether the lot must be reanalyzed.

4. If data points for fewer than two-thirds of the control analytes are classified as in control (more than one-third meet one of the out-of-control conditions), the method is considered to be out of control and all work on that method (including sample preparation) must cease immediately. No data for environmental samples in that lot may be reported. Efforts must be initiated to determine the cause of the problem. If the problem is instrumental or specific only to preparation of that lot, samples prepared after the out-of-control situation occurred may be processed after the instrumental system is repaired and recalibrated, provided that holding times are not exceeded. If no specific cause can be assigned, the instrument should be recalibrated and all samples prepared subsequent to the last in control lot should be prepared. In any case, the out-of-control lot must be re-analyzed. The out-of-control situation and corrective actions taken must be fully documented. Each point must be annotated with a reference to the investigation and to the disposition of samples and results.

5. The establishment of overall method control for analyses may not be accurate for describing a particular analyte(s). For analyses in which control cannot be established for certain control analytes (i.e., loss of surrogate due to volatility), such analyte results may still be deemed as out of control even though the method is considered in control. The evaluation of control in such instances will be handled on a case-by-case basis.

If a lot is still out of control after re-analysis, all method-related activities will cease immediately. A detailed laboratory-wide investigation will be conducted to isolate and correct faulty operations. Sample security, integrity of standards, reagents, glassware, laboratory notebooks, instrument performance, and adherence to certified methods should be included in the investigation.

An out-of-control situation for R control charts may be indicated by:

- A value above the UCL or classified as an outlier by testing
- A series of five consecutive points going in an upward direction
- A cyclical pattern of control values
- Two consecutive points between the UWL and UCL

Whenever one of the conditions is detected, the analyst and LQAC must investigate. The control and warning limits for the three-point moving average charts for Class 1 analyses are not

intended for method control by themselves but are evaluated in conjunction with results from spikes at the high concentration. However, if the spiked concentration is not detected within acceptable control limits in two consecutive lots, the method will be considered out of control. Data falling outside control chart limits may indicate quality problems and should be investigated to identify serious problems. The entire analytical process will be investigated and corrected to achieve the required sensitivity. All actions taken will be documented.

7.4 EXTERNAL QC SAMPLE CONTROL

External QC samples are samples collected during field operations or generated to evaluate transportation induced contamination of investigate samples. External QC samples include the following categories of samples and designated purposes.

Duplicate Samples

Collocated Samples - Independent samples collected such that they are equally representative of the parameter(s) of interest at a given point in space and time. When collected, processed, and analyzed by the same organization, these samples provide intralaboratory precision information for the entire measurement system, including sample acquisition, homogeneity, handling, shipping, storage, preparation, and analysis. Can also be used to estimate the overall precision of a data collection activity.

Replicate Samples - Samples that have been divided into two or more portions at some step in the measurement process. Each portion is then carried through the remaining steps in the measurement process. A sample may be replicated in the field or at different points in the analytical process. For field replicated samples, precision information would be gained or homogeneity (to a lesser extent than for collocated samples), handling, shipping, storage, preparation, and analysis. For analytical replicates, precision information would be gained on preparation and analysis.

Trip Blanks

Trip blanks generally pertain to VOC samples only. These samples are prepared by the laboratory using analyte-free water prior to the sampling event in the actual sample bottles and are kept with the investigative samples throughout the sampling event, then packaged for shipment to the laboratory for analysis with other samples. One trip blank should be included in each shipping container. At no time after preparation are sample bottles opened before they reach the laboratory.

Field Blanks

Field blanks are samples prepared from water used during the decontamination process. Samples are prepared by pouring deionized water used in decontamination into the

appropriate sample vial and analyzing for most semivolatile or volatile organics and metals. This allows the QAC to remove the contribution due to deionized water contamination from that which may be attributed to insufficient decontamination procedures, as determined using rinse blanks.

Rinse Blanks

Rinse blanks are defined as samples obtained by running analyte-free deionized water through sample collection equipment (bailer or pump) after decontamination and placing it in the appropriate sample containers for analysis. These samples will be used to determine the adequacy of field decontamination procedures. Using the above definition, soil rinse blanks could be called rinsate samples. These will be included in the sampling program on a daily basis.

Duplicate samples will be collected for 10 percent of the investigative samples or one per day, whichever is more frequent. Trip blanks will accompany each shipment container or set of coolers to be sent on a given day. Trip blanks will be analyzed only when volatile organics are specified as target compounds. Rinse blanks and field blanks will be collected once per day or for 5 percent of the investigative samples, whichever is more frequent.

Accuracy and precision of sample data will be monitored on a routine basis. Procedures for evaluating accuracy and precision are described below for each QA/QC sample type.

The evaluation procedure for blanks is a qualitative review of the analytical data reported by the laboratories. The procedure for assessing blank samples will be as follows:

1. Tabulate data from the blank samples.
2. Identify blank samples that exhibit detectable concentrations of analytes in the sample.
3. If no analytes are detected in any blank samples, the tables are ready for entry into the appropriate report.
4. If chemicals are found in blank samples, the compound(s) and concentration(s) will be reported and the field data for that period of time will be assessed for potential problems with data interpretation. No data will be removed from the data base on the basis of analytes being detected in blank samples. Appropriate notation, however, will be made in the data base reports.

The procedure for assessing duplicate (collocated and replicate) samples will be as follows:

Tabulate duplicate data and calculate the Duplicate Sample Agreement (DSA) percent, Replicate Sample Agreement (RSA) percent, and Relative Percent Difference (RPD) as shown below for each duplicate pair:

$$DSA(\%) = \frac{X_1}{(X_1 - X_2)/2} \times 100\%$$

$$RSA(\%) = \frac{R_1}{(R_1 - R_2)/2} \times 100\%$$

$$RPD(\%) = \frac{|X_1 - X_2|}{X} \times 100\%$$

Where: X_1 or R_1 = concentration for sample 1 of duplicate/replicate

X_2 or R_2 = concentration for sample 2 of duplicate/replicate

X = average of sample 1 and 2 or replicate 1 and 2

The procedure for assessing performance samples will be as follows:

Tabulate spike sample data and calculate the Spiked Sample Recovery (SSR) percent as shown below for each sample:

$$SSR(\%) = \frac{T - \overline{X}}{A} \times 100\%$$

where:

T = total concentration found in spiked sample

X = original concentration in sample prior to spiking

A = actual spike concentration added to sample

7.5 DATA REDUCTION

Data reduction is the process in which analytical and field data are evaluated in terms of precision, accuracy, representativeness, completeness, and comparability. This is a multiphased task that involves data collection, validation, and the spatial significance of data relationship to program objectives. Two basic types of data must be reduced during the project. Field measurements and analytical data will be evaluated in terms of precision, accuracy, completeness, representativeness, and comparability. In essence, data reduction is the application of the QA

Plan to analytical and field data collected during a project. This reduction process affects the ultimate usability of program results and will include the following data processing steps:

1. Data collection (laboratory or field)
2. Calculation of preliminary results
3. Internal and external validation of results
4. Accuracy of reporting procedures
5. Plotting and spatial evaluation of analytical results
6. Identification of project critical data points
7. Re-evaluation of program results relative to initial program objectives
8. Generation of a report detailing data usability and technical utility

Data that have been reduced are in final form until more work is performed or new information becomes available that modifies the effect of an element in the reduction process. A key element in data reduction is consistency. This requirement for consistency in reduction steps is the primary purpose behind the development and implementation of a quality assurance program.

7.6 DATA VALIDATION

Field measurement data validation will be performed by the QAC or Quality Assurance Field Supervisor. Validation will be performed by checking procedures utilized in the field against the checklisted requirements shown in Appendix C of this QA Plan and project-specific requirements presented in the appropriate project plans.

The following reporting requirements for field data will be followed:

- pH - Field measurements will be reported to the nearest $\pm$0.1 pH value
- Electrical conductivity - Field measurements will be reported to two significant figures
- Water levels - Measurements will be repeated until at least two are documented to be in agreement to the nearest 0.02 foot
- Soil sample depths - Tape measurements will be made to the nearest 0.1 foot; measurements made by known lengths of drill string will be made to the nearest 0.5 foot

- Elevations of sampling sites

  - Permanently marked measuring points for all monitoring wells will be surveyed to the nearest 0.01 foot and referenced to mean sea level

  - Approximate elevations of all other nonsurveyed sampling sites will be determined to the nearest 1.0 foot

- Locations of sampling sites - Locations of soil and sediment sampling sites, monitoring wells, surface water, waste, biota, air, and sampling sites, and piezometers will be surveyed to the nearest 1.0 foot

- Lithologic descriptions - Sample descriptions will be consistent with the Unified Soil Classification System

Each laboratory that uses a PMRMA-certified method is required to operate a formal QC program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data accuracy and precision. The laboratory must maintain records to document the quality of data that are generated. Ongoing data quality checks are to be compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method being used. When results of sample spikes indicate atypical accuracy or precision, a QC check standard must be re-analyzed to confirm that the measurements were performed in an in-control mode of operation. Method performance requirements must have been met prior to the analysis of any samples. Performance evaluation control chart data will accompany the data transfer files to which they apply.

Calculations performed by the laboratory for reporting chemical concentrations of analytes will be performed according to procedures specified in the appropriate certified method.

The laboratories will perform analytical data reduction and in-house validation under the direction of the LQAC. The LQAC will be responsible for assessing data quality and advising appropriate section supervisors and the QAC of data that are rated "unacceptable" or of other notations that would caution the data user of possible unreliability. Data reduction, validation, and reporting by the laboratory will be conducted as follows:

1. Raw data produced by the analyst will be turned over to the respective section supervisor.

2. The section supervisor will review the data for attainment of QC criteria outlined in this QA Plan.

3. Upon acceptance of the raw data by the section supervisor, a computerized report will be generated and sent to the LQAC.

4. The LQAC will complete a thorough audit of 100 percent of every report for consistency.

5. The LQAC and section supervisor will determine whether re-analysis of any sample is required.

6. Upon acceptance of the preliminary results by the LQAC, transfer files will be generated and the data will be sent to HLA.

The LQAC will conduct an evaluation of data reduction and reporting by the laboratory. This evaluation will consider the analytical sequence, calculation sheets, document control forms, blank data, duplicate data, and recovery data for QC samples and calibration standards. The material will be checked for legibility, completeness, correctness, necessary dates, initials, and signatures. Assessment of analytical data will include checks for consistency by assessing the comparability of duplicate analyses, the comparability to previous data from the same sampling location (if available), adherence to accuracy and precision QA plan criteria, transmittal errors, and anomalously high or low parameter values. The results of this check will be reported to the QAC, noting any discrepancies and their effect on acceptability of the data.

The laboratory LQAC will also review at least the following analytical procedures and instrument performance criteria:

Organics Analysis

- Data completeness
- Sample holding time
- GC/MS tuning and mass calibration
- GC/EC calibration and column conditions for pesticides/PCBs
- Blanks
- QC sample recoveries
- Control charts

- Compound identifications
- Compound quantifications

Inorganics Analysis

- Data completeness
- Sample holding time
- Instrument calibration
- Blanks
- Interference check sample analysis
- IDL and spike sample recoveries
- Laboratory control sample analysis
- Standard addition results
- Quarterly verification of instrument parameter report
- Appropriate concentration units
- Appropriate significant figures
- Samples that exhibit carry-over effects
- Extraction efficiency
- Spectral interpretation
- GC/EC confirmation
- Inconsistency

Laboratory records and data package requirements will be checked to assess completeness of the data package.

The following is a brief description of the validation steps that will be used by the QAC or designated representative to independently review validated laboratory data.

1. Compile a list of all investigative samples
2. Compile a list of all QC samples, including:
   - Field blanks

- Trip blanks
- Laboratory blanks
- Laboratory duplicates
- Performance samples
- QC samples

3. Review chain-of-custody documents for completeness and correctness
4. A data summary will be prepared and will include:
   - Results
   - Sample media identification
   - Unexpected results
   - Common laboratory contaminants
   - Unusual concentrations and identifications
   - Samples in which dilution was unnecessary

Despite all efforts to achieve the objectives of the QA plan, the potential for error exists in laboratory chemical analyses and in the data reporting process. Every reasonable effort will be made to compare and double-check data reported from the laboratory and data entered into the IRDMS.

7.7 REPORTING REQUIREMENTS

All numerical results will be reported in terms of concentration in the environmental sample. Resultant found concentrations submitted for entry into the PMRMA IRDMS must remain unadjusted before being reported to PMRMA. Correction factors (e.g., accuracy, percent moisture, and dilution factor) are maintained separately in the IRDMS. All data are collected during periods when calibration and control systems were used. As described earlier, only concentrations measured within the certified range, prior to correction, are reported. Specific instructions are provided in the IRDMS User's Guide regarding the coding of entries.

In reporting results, rounding to the correct number of significant figures occurs only after all calculations and manipulations are completed. The number of figures warranted by the analytical technique should be used in pre-reporting calculations. Premature rounding can significantly affect the final result. The method blank results are subtracted on an instrument level prior to calculation of a found concentration. Each analytical method describes the correct procedure for using method blank results.

Class 1, Class 1A, and Class 1B Certified Methods

All uncorrected values less than the CRL, including no response, are reported as "less than" the reporting limit.

Class 1 and 1B Methods

If results for an analyte were obtained using the method exactly as tested, without dilution, the analyte concentration in the sample may be reported to three significant figures. If dilution was required for a particular analyte, the result is reported to only two significant figures, reflecting the fact that total method performance was not demonstrated at that concentration during certification. Noncertified analytes, either identified or unknown, are reported by retention time in terms of positive or negative. Such results will be brought to the attention of the PMRMA Project QA Officer for evaluation on a case-by-case basis.

Class 1A Methods

Results for certified analytes (target and surrogate) are reported to two significant figures if the method was used without dilution. Results obtained after dilution and results of screening for noncertified analytes are reported to only one significant figure.

Class 2 Certified Methods

The results of Class 2 certified methods are not adjusted for dilution or accuracy. The results for samples analyzed by Class 2 certified methods are measured in relation to the CRL

(two significant figures) and reported as "less than," "equal to," or "greater than" the CRL. A tested concentration range is not applicable because only the CRL concentration is tested.

Appropriate documents will be prepared and distributed to summarize the field activities performed and the results obtained. These reports, to the extent applicable, will include the following:

- Presentation of results
- Summaries of field data from field measurements such as well discharge, water levels, and water-quality parameters
- Field location of sampling points
- Lithologic description logs
- Schematic diagrams of well construction for monitoring wells

Raw data from field measurements and sample collection activities will be maintained in the project files and will be presented in the appropriate reports. If data have been reduced or summarized, the method of reduction will be documented in this report.

8.0 INSTRUMENT MAINTENANCE

Procedures described in this section pertain to the calibration, maintenance, and operation of field and laboratory equipment. This section establishes procedures for maintaining test and measurement equipment used to conduct analyses. The calibration policies and procedures set forth apply to all tests and measuring equipment. Test and measurement instruments to be used fall into two general categories: those that are calibrated prior to each use and those that are calibrated on a scheduled periodic basis.

8.1 FIELD INSTRUMENT MAINTENANCE

A variety of instruments, equipment, and sampling tools will be used to collect data and samples and to monitor field conditions. Proper calibration, maintenance, and use of instruments and equipment is required to ensure quality of field data collected.

8.1.1 Inspection

All instruments and equipment purchased or used will be inspected to ensure that each item meets and performs to manufacturer specifications and project specifications. Instruments meeting these requirements are given a serialized number and made available for site use. Instruments and equipment not meeting program requirements are labeled and are withheld from field use. Such instruments and equipment are not available for use until they can be modified or repaired to meet the site requirements.

8.1.2 Operating Procedures

The calibration, maintenance, and operating procedures for field instruments, equipment, and sampling tools are documented in the owner/operator manuals supplied by the manufacturer. These manuals provide manufacturers' instructions and include specifications and criteria for calibration, maintenance, and operation.

8.1.3 Field Equipment Calibration

Each item of equipment used in field activities is calibrated at a frequency specified in the owner/operator manuals provided by the manufacturer. These specifications and procedures are provided in the FOP and contain at a minimum:

- Equipment identification number
- Control number
- Calibration schedule and frequency
- Equipment specifications5Xapplicable)
- Equipment necessary to accomplish calibration
- Procedure for calibration

Equipment calibration is recorded daily or as required by the calibration schedule. Calibration is recorded by HLA field personnel in bound field notebooks. Information recorded includes the following:

- Date of calibration
- All data pertaining to the calibration procedures
- Initials of analyst performing calibration
- Adjustments made to the equipment prior to and following calibration
- Record of equipment failure or inability to meet specifications

If the calibration schedule is not adequately maintained or accuracy, as reported in the operations manual, cannot be attained, that instrument is identified and unavailable for use until repaired so that specifications are attained.

General calibration requirements for field equipment used during this project are described below and are consistent with manufacturers' operations manuals.

Organic Vapor Analysis

The portable gas analyzers currently identified as being available for onsite use during field operations are HNu Model P1-101 photoionization analyzers. Equivalent instruments may also be used during the investigation. Calibration procedures will be followed in accordance with the factory-supplied instruction manual.

Water-Level Measurements

Electrical sounder calibration:	Check against steel surveyor's tape prior to use.
Graduated steel tape calibration:	Manufacturer-supplied temperature correction will be applied if applicable for field conditions.
Pressure transducer calibration:	Factory-calibrated once, in-house calibration check with water columns prior to aquifer tests, and weekly field checks against steel tape or electrical sounder during use.

pH Measurement

Digital pH meter calibration: (Beckman Model 021 or equivalent)	Factory- or laboratory-supplied buffer solutions will be renewed daily and used prior to and following each use. Temperature correction will be applied during measurement.

Specific Conductance

Electric conductivity meter calibration: (YSI Model 51B or equivalent)	Factory-calibrated annually, calibrated prior to each use using laboratory-supplied KCl standard, and temperature correction will be applied during measurement.

Water Temperature

Mercury thermometer calibration:	Factory-calibrated once and checked at least annually.
Temperature meter calibration:	Calibrated weekly against a mercury thermometer.

8.1.4 Maintenance

Each item of equipment used in field activities is maintained to specifications presented by the manufacturer. The Field Activities Manager will be responsible for performing routine maintenance and will have available tools and spare parts to conduct routine maintenance. Main-

tenance items that cannot be performed by the Field Activities Manager will be performed by a person certified or trained to repair the instrument.

Procedures for maintaining instruments are consistent with manufacturers' operations manuals. Instruments will be calibrated to proper specifications following maintenance to ensure proper completion of the maintenance procedure.

A record of maintenance, including a description of specific activities performed, will be made in the field log notebook. This notebook is kept in the site trailer with the instrument. Data recorded in the logbook are similar to the data recorded for calibration.

If the equipment or instrument cannot be maintained to the manufacturer's specifications or cannot be properly calibrated, it will be returned to the manufacturer or other repair facility for proper maintenance and/or repair. Once received back from the manufacturer, the instrument will be checked for compliance to project specifications before being returned to routine field use.

8.2 LABORATORY INSTRUMENT MAINTENANCE

Laboratory preventative maintenance programs are designed to prevent instrument breakdown during the analyses of investigative samples. Such programs are directed at critical instrument pathways such as calibration of instruments and maintaining physical conditions consistent with those used during the certification process. PMRMA analytical tests are based on the ability to perform an analytical testing sequence of events under the identical conditions with a high degree of reliability. For this reason a preventative maintenance program for PMRMA IR studies must address instrument service and the absolute physical condition of any critical measurement elements involved in the analytical sequence.

Instruments requiring calibration must be assigned a maintenance log number or record number. This label will be used by maintenance personnel and include a description of the instrument, manufacturer, model number, serial number, data of last calibration or maintenance, signature of maintenance person, and date when next service check is required.

A maintenance log will be kept for each instrument or measuring device to indicate the history of maintenance performed on that piece of analytical equipment. Measuring devices too small to receive maintenance labels must still be accompanied by a maintenance or calibration logbook in the area where they are used. Chemical calibrations are not considered absolute calibrations and are therefore not addressed in standard maintenance procedures. Absolute calibration procedures must be kept along with the instruments that require this type of calibration. Chemical calibrations are useful as indicators of instrument problems or calibration standard degradation and may therefore be used to qualitatively identify non-routine maintenance requirements. Maintenance of absolute calibrations is required for many types of laboratory equipment. This type of equipment includes but is not restricted to the following types of laboratory apparatus.

- Instrument recording units such as chart recorders that must be absolutely calibrated for a certain signal or electronic input.
- Flow controllers or instruments that must remain constant to assure instrument stability. Flow controllers on fume hoods to maintain the safety of laboratory personnel and maintain a contamination free analytical environment.
- Temperature sensors of all types both internal to instrumentation and external such as thermometers used to maintain constant sample storage and glassware decontamination conditions.
- Syringes and pipettes used to introduce standards and investigative sample extracts and all measurement apparatus for aliquot fractioning.

Instruments or measuring devices that have not received maintenance or routine calibration must not be used to perform project related work. If an instrument requires absolute calibration based on historical or manufacturer's recommended guidelines, it will be removed from service until such maintenance can be performed. In such cases a physical label must be placed in an obvious location on the instrument so as to prevent accidental use of uncalibrated equipment. Such instruments should be removed from locations in which accidental use is easily performed. Calibration of critical path instrumentation and routine instrument maintenance must be traceable through instrument tags or maintenance log books. Absolute calibrations must be performed

using the appropriate and accepted absolute standard reference. For balances NBS-certified Class "S" weights should be used to calibrate instrumentation. Use of in-house Class "S" weights is acceptable for daily calibration, but a yearly certification by an external inspector should also be performed. Similarly, an NBS certified thermometer should routinely be used to calibrate all temperature gauging devices. In all cases when calibration is performed it must be easily traceable in the maintenance log books. Instruments that do not require certified calibration such as instrument flow rates should be checked routinely. For example GC/MS and GC flow rates should be checked daily or on a regular basis utilizing a bubble meter to assure consistency of sample introduction.

Each laboratory must maintain a standard procedures manual for preventative maintenance of critical and routinely used instrumentation. A laboratory must provide these standard procedures to each analyst so if maintenance is required the appropriate source of assistance is readily available. Each laboratory must employ a qualified maintenance person or be covered by a maintenance contract for all project required instruments.

The maintenance program employed by a laboratory should be targeted at minimizing instrument downtime. The substitution of calibration protocol described in this document must be reviewed and approved by PMRMA prior to the initiation of program activities.

9.0 RECORDKEEPING

The aim of recording and documenting the steps involved in the analysis of environmental samples is to ensure that the data acquired are of the highest technical merit. The technical utility of analytical data in legal action is directly dependent on the availability and correctness of supporting documentation. The results reported for a carefully performed analytical test are only as good as the documentation that supports that result. Documentation for analytical results provides lasting evidence of data validating, traceability, sample security (integrity), representativeness, and retrievability.

Documentation of sample collection and analysis will require full chain-of-custody procedures or the equivalent. All such documentation must be available for inspection during and after the completion of project related analyses. Documentation must be kept in pre-numbered, bound logbooks to provide a chronological record of the sequence of events or steps taken during the program. Reference documents must include a table of contents according to time, type of analysis performed, sample matrix, and signature of the analyst. All logbook entries must be performed using permanent ink. Correction will be performed by drawing a single line through entries to be corrected, entering the correct information, and initialing and dating the change.

Computerized logging systems may not be used to replace bound logbooks unless computerized entries are permanently attached to the logbook and paginated appropriately. A separate logbook must be maintained for the RMA program samples. Logbooks kept for multiple installation studies are unacceptable. This does not apply to any routine logbooks kept to monitor overall laboratory performance or instrument maintenance because documentation procedures vary from one laboratory to another. All standard documentation procedures to be employed during the performance of project-related activities must be available for review by the program QAC and PMRMA prior to the start of work.

9.1 FIELD ACTIVITIES DOCUMENTATION

The above guidelines apply to all steps involved in the analysis of environmental samples starting with the field collection logbook to the final disposal or storage of the processed samples. All field or sampling logs will be completed in waterproof ink on logbook paper that is water resistent. The field sampling logbook must contain information to distinguish each sample from any other investigative or external QC sample. This information must be clearly formatted in a consistent manner as to be easily identified and reviewed and include:

- Identification of the PMRMA project for which collection is being performed
- Unique sequential sample number or designation
- Sample type or matrix
- Sample depth
- Date and time of sampling
- Detailed description of sample location
- Approved sampling method used
- Preservation technique
- Order of steps used, such as filter and then preserved
- Analytes or interest
- Volume and container size/type
- Detailed description of sample character (i.e., grain size, sorting, color, etc.)
- Results of field measurements, such as volume of water purged prior to sampling, temperature, conductivity, and pH
- Names and signatures of sampling personnel
- Date of sample shipment, number of shipping containers, volume of each sample type, and carrier used

Environmental samples collected during field activities must be carefully labeled and labels reviewed for legibility by the Field Quality Assurance Coordinator (FQAC). Each sample label will be reviewed and analyzed by the FQAC prior to shipment. Labels will be attached to sample

containers using water resistent glue and secured with waterproof Scotch tape. Labels will be filled out just prior to sampling and include the following minimum information.

- Installation or location ID name
- Unique sequential field sample number
- Sampling data
- Analyses requested
- Preservative/filtration
- FQAC's and sampler's initials

All relevant paperwork such as chain-of-custody forms and airbills will then be sent to the laboratory and stored in the final evidence or installation restoration analyses record file. A copy of the field logbook and chain-of-custody forms will then be submitted to the HLA QAC. Forms will be reviewed, as well as entered, into the automated sample tracking system and office project files. Field data sheets will also be completed and signed for each field measurement and copies provided to the QAC and Project Manager for filing into the project record files.

9.2 LABORATORY ANALYSIS DOCUMENTATION

Sample shipment to the laboratory will be performed by an authorized courier. This courier will sign-off on the airbill to document the path taken by the samples in reaching their destination. The sample custodian for the laboratory will then sign the airbill as received and maintain a copy of it for the laboratory project file. The chain-of-custody will then be removed from the cooler and the contents checked for breakage, completeness, and temperature. If the integrity of any sample is evidenced as being threatened the HLA QAC will be notified immediately. If all samples appear satisfactory, the samples will be logged into a bound installation specific logbook that will include at least the following information.

- Field sample number
- Data of arrival
- Observations concerning sample integrity

- Analysis requested
- Sample splits performed, if necessary
- Volume of sample received
- Number of containers per sample received
- PMRMA sample ID numbers including the appropriate laboratory QC sample numbers utilizing the format specified in this document

9.3 PMRMA SAMPLE IDENTIFICATION NUMBERS

Reporting to the PMRMA IRDMS requires that each analytical aliquot of a sample be assigned a six-character sample identification number, comprised of two three-character designations. The first three characters are letters that indicate the analytical lot. Analytical lot designations will be based on the number of samples that may be simultaneously processed through the rate limiting step of the analytical method; the rate of sampling or number of samples shipped to the laboratory at the same time do not determine lot size. A different lot designation is used for each analytical method. For multi-analyte methods, the lot designation is the same for each analyte in a single sample fraction. A different lot designation is used for each lot analyzed by a particular method. If the contractor laboratory used an internal numbering system, a correlation to the PMRMA sample identification number shall be provided in the log-in-book.

The last three characters of the six-character identification number are sample analysis numbers assigned in sequential order to indicate the instrumental order of analysis within a lot.

Three examples of PMRMA sample identification number assignments are provided below:

1. Six samples are received by a particular laboratory for 2,4,6-trinitrotoluene (2,4,6-TNT), 2,4-dinitrotoluene (2,4-DNT), trichloroethylene (TRCLE), and tetrachloroethylene (TCLEE) analyses. 2,4,6-TNT and 2,4-DNT are analyzed simultaneously with the same method; TRCLE and TCLEE are also analyzed simultaneously using another method. For this laboratory, each method (Class 1B) has a maximum lot size of 8 samples. Therefore, the assignments could be made as shown in Table 9.1.

2. Nine samples are received for cadmium, Cd, chromium, Cr, nickel, Ni, and lead, Pb, analyses. If the laboratory is using a multi-analyte technique, such as inductively coupled plasma emission spectroscopy (ICAP), all samples could be assigned to the same lot and each analyte in the same sample would have the same sample number. If the laboratory is using a single-analyte method, such as atomic absorption spectroscopy (AAS), each aliquot is a different lot that could be designated as shown in Table 9.2.

Table 9.1: PMRMA Sample Identification Numbers for Example 1

	Method 1		Method 2	
	2,4,6-TNT	2,4-DNT	TRCLE	TCLEE
Sample 1	AAA 002	AAA 002	AAB 001	AAB 001
Sample 2	AAA 003	AAA 003	AAB 003	AAB 003
Sample 3	AAA 005	AAA 005	AAB 004	AAB 004
Sample 4	AAA 006	AAA 006	AAB 005	AAB 005
Sample 5	AAA 007	AAA 007	AAB 007	AAB 007
Sample 6	AAA 008	AAA 008	AAB 008	AAB 008
Sample Blank	AAA 001	AAA 001	AAB 006	AAB 006
Spiked QC-High	AAA 004	AAA 004	AAB 002	AAB 002

Note that analytes by the same method have the same exact sample number. Sample number sequence need not be the same for the two methods.

Table 9.2: PMRMA Sample Identification Numbers for Example 2

	Multi-Analyte Method			
	Cd	Cr	Ni	Pb
Sample 1	AAC 001	AAC 001	AAC 001	AAC 001
Spiked QC-High	AAC 002	AAC 002	AAC 002	AAC 002
Sample 2	AAC 003	AAC 003	AAC 003	AAC 003
Sample 3	AAC 004	AAC 004	AAC 004	AAC 004
Method Blank	AAC 005	AAC 005	AAC 005	AAC 005
Sample 4	AAC 006	AAC 006	AAC 006	AAC 006
Sample 5	AAC 007	AAC 007	AAC 007	AAC 007
Spiked QC-Low	AAC 008	AAC 008	AAC 008	AAC 008
Sample 6	AAC 009	AAC 009	AAC 009	AAC 009
Spiked QC-High	AAC 010	AAC 010	AAC 010	AAC 010
Sample 7	AAC 011	AAC 011	AAC 011	AAC 011
Sample 8	AAC 012	AAC 012	AAC 012	AAC 012
Sample 9	AAC 013	AAC 013	AAC 013	AAC 013

	Single-Analyte Method			
	Cd	Cr	Ni	Pb
Method Blank	AAD 001	AAE 012	AAF 004	AAG 002
Sample 1	AAD 002	AAE 001	AAF 001	AAG 003
Sample 2	AAD 003	AAE 002	AAF 003	AAG 004
Spiked QC-High	AAD 004	AAE 009	AAF 007	AAG 001
Sample 3	AAD 005	AAE 004	AAF 005	AAG 005
Sample 4	AAD 006	AAE 005	AAF 006	AAG 006
Sample 5	AAD 007	AAE 007	AAF 008	AAG 007
Sample 6	AAD 008	AAE 008	AAF 009	AAG 008
Spiked QC-High	AAD 009	AAE 006	AAF 002	AAG 012
Sample 7	AAD 010	AAE 010	AAF 011	AAG 009
Sample 8	AAD 011	AAE 011	AAF 012	AAG 010
Sample 9	AAD 012	AAE 013	AAF 013	AAG 011
Spiked QC-Low	AAD 013	AAE 003	AAF 010	AAF 013

For this laboratory, maximum lot size for both the ICAP and AAS methods (Class 1) is 25 samples.

3. Ten samples are received by a particular laboratory for 2,4,6-TNT, 2,4-DNT, and mercury, Hg, analyses. 2,4,6-TNT and 2,4-DNT are analyzed simultaneously by the same method (Class 1) and maximum lot size in this laboratory is 12 samples. Maximum lot size for the mercury method (Class 1) in this laboratory is 20. The laboratory could assign identification numbers as shown in Table 9.3. Note that all samples for 2,4,6-TNT and 2,4-DNT were collected and shipped to the laboratory together; however, the lot designation is not the same for all these samples due to maximum lot size limitation.

9.4 ANALYTICAL RECORDS

As previously detailed in this document, SARMs and QC analytical reference materials must be tracked in a bound logbook. This record must include date of receipt, source, purity, label information, storage conditions, and expiration date. This logbook should also maintain a record of reference material performance. Similarly, reagents used in sample preparation must also be logged and their performance tracked. A standard preparation logbook will also be maintained for each type of QC sample (i.e., VOAs, BNAs, pesticides). This logbook will contain details concerning QC sample preparation and will include the following types of information:

- Solvent used
- Source of stock
- Concentration of stocks used in preparation
- Final concentration of QC sample
- Dilutions performed
- Solvent lot number
- CAS number of each analyte
- Initials of chemist preparing the solution
- Date of preparation/expiration

Every instrument utilized in the RMA program will have an associated instrument logbook. This logbook will be maintained at the instrument and will not under any circumstances be removed from the instrument location. This logbook will be bound and project-specific if

Table 9.3: PMRMA Sample Identification Numbers for Example 3

	Method 1	
	2,4,6-TNT	2,4-DNT
Sample 1	AAR 001	AAR 001
Method Blank	AAR 002	AAR 002
Sample 2	AAR 003	AAR 003
Spiked QC-High	AAR 004	AAR 004
Sample 3	AAR 005	AAR 005
Spiked QC-High	AAR 006	AAR 006
Sample 4	AAR 007	AAR 007
Spiked QC-Low	AAR 008	AAR 008
Sample 5	AAR 009	AAR 009
Sample 6	AAR 010	AAR 010
Spiked QC-Low	AAS 001	AAS 001
Sample 7	AAS 002	AAS 002
Spiked QC-High	AAS 003	AAS 003
Sample 8	AAS 004	AAS 004
Method Blank	AAS 005	AAS 005
Sample 9	AAS 006	AAS 006
Spiked QC-High	AAS 007	AAS 007
Sample 10	AAS 008	AAS 008

	Method 2
	Hg
Sample 1	AAQ 001
Sample 2	AAQ 002
Spiked QC-High	AAQ 003
Sample 3	AAQ 004
Sample 4	AAQ 005
Spiked QC-High	AAQ 006
Sample 5	AAQ 007
Sample 6	AAQ 008
Method Blank	AAQ 009
Sample 7	AAQ 010
Sample 8	AAQ 011
Spiked QC-Low	AAQ 012
Sample 9	AAQ 013
Sample 10	AAQ 014

possible. All daily instrument operations will be recorded in this logbook to allow for the reconstruction of the daily operating sequence. Each entry to this logbook will be signed by the analyst responsible for that injection or maintenance procedure. All instrument activities such as reanalyses and instrument maintenance time will be recorded in this logbook. Under no circumstances will entries be deleted or added to this logbook. Each entry for sample analysis will include but not be limited to the following:

- Data of analysis
- Analysis time of injection
- Test name
- Project ID
- Sequential number
- Associated calibration and method blank/QC samples
- Analyst's signature
- Amount of sample injected
- Comments concerning instrument performance

When automated data acquisition systems are used, reference to the data file for each standard or sample will be recorded.

Hard-copy output (such as chromatograms and integrator tapes) from instruments will be labeled with analyst's name, analysis time, test name, sample number, reference to the calibration curve used or quantification, and reference to the logbook where analytical activities were recorded; the identity of chromatographic peaks will also be noted. The pages are maintained with the lot data packages.

An individual instrument maintenance logbook is assigned to each instrument on a permanent basis and is not turned over to PMRMA after project completion.

The results for samples analyzed in support of PMRMA IR projects will be entered in the PMRMA IRDMS. Specific instructions are provided in the IRDMS/User's Guide supplied to

each contract laboratory. Specific information on data management is contained within the project Data Management Plan.

10.0 AUDITS

Audits are a systematic evaluation to monitor the performance of the total measurement system. Program laboratory and field audits are separated into external and internal audit functions. External audits are those audits performed by the PMRMA Officer. Internal audits are conducted by the HLA QAC or his designated representative. All program audits will focus on confirming the field and laboratory's efficiency at implementing sampling and analytical requirements described in this QA Plan.

10.1 EXTERNAL AUDITS

External audits conducted by PMRMA will focus on the ability of the proposed QA Plan to meet project objectives. External auditors will discuss any program weaknesses or discrepancies in relationship to the established PMRMA Chemical QA Plan directives or good laboratory practices already in operation at a contractor laboratory. During external audit visits the PMRMA representative will fill out the standard initial visit audit forms provided in the PMRMA Chemical QA Plan (August 1989). Copies of this completed standard form will be provided to the HLA Project Manager, the HLA QAC, and the LQAC. All audits will focus on systematically evaluating the quality of operations of individual laboratory systems. The efficiency of any system is inherently dependent on the qualification of laboratory personnel, proper management guidelines, the availability or knowledge of laboratory personnel in relationship to PMRMA protocols, a well established document/sample tracking procedure, an efficient QA/QC program, and an error-free, easy to operate report generation system. If during the initial external audit serious deficiencies are found, copies of the report may be forwarded to the Contracting Officer at Procurement for official documentation and action. Subsequent to project initiation a PMRMA representative may visit the analytical laboratory or sampling location to evaluate the effective implementation of the project QA Plan objectives. Analyses in progress will be open for inspection at this time and discrepancies identified in a second audit report. This second audit report will be provided to the contractor laboratory and PMRMA management

as required. External audits may be scheduled or unscheduled and need not be limited to two visits if program inconsistencies demand a closer level of external control.

10.2 INTERNAL AUDITS

Internal audits will be conducted by the LQAC or the HLA QAC. Internal audits will specifically target discrepancies found during external audits and include a detailed review of laboratory and field procedure using a step-by-step approach. Internal audit procedures will be performed in such a way that all phases of analysis are reviewed sequentially in detail. Field QC audits will include a checklist review consistent with the PMRMA Chemical QA Plan (Appendix F) and an onsite review of field notebooks, photographs, sampling locations, collection technique, filtering, preservation, sample labels, chain-of-custody forms, shipment of samples, field measurements, and completion of field measurement forms.

Internal laboratory audits will include a systematic review of laboratory facilities, equipment, training procedures, recordkeeping, data validation, data management, reporting, and QA checks. During this review the HLA QAC will carefully review the standard preparation procedures, calibration logs, tuning logbooks, raw data collection, and confirmation steps. The QAC will attempt to identify laboratory discrepancies with the QA Plan, overall deficiencies, and inappropriate laboratory practices. Based on the audit, corrective action will be suggested. If the corrective action required is sufficiently serious, project work will be stopped until corrective action has been performed and a work restart order will be authorized by the HLA QAC. Audits will be performed before and during the analysis of project samples. Audits may be announced or unannounced. The audit report format will be consistent with the PMRMA checklist and include appropriate features from the EPA CLP prebid audit reporting format. Copies of all internal audit reports will be supplied to PMRMA for review and comment. Corrective action forms or requirements will be supplied to the LQAC in addition to the formal audit report.

11.0 CORRECTIVE ACTION

11.1 LABORATORY CORRECTIVE ACTION

Corrective action must be implemented whenever audit or QC sample results indicate that a problem that will jeopardize the integrity of investigative sample results exists. Corrective action or stop-work memoranda may be authorized by PMRMA, HLA Project Manager, HLA QAC, LQAC, or Laboratory Section Supervisor. Analysts who suspect that an out-of-control situation may exist are obligated to inform their Section Supervisor of the problem before stopping work. If previously reported data are affected by a situation requiring corrective action or if the corrective action will impact the program budget or schedule, the action should directly involve the HLA Project Manager and PMRMA.

Corrective actions are of two kinds:

1. Immediate, to correct or repair nonconforming equipment and systems. The need for such an action will most frequently be identified by the analyst as a result of calibration checks and QC sample analyses.
2. Long-term, to eliminate causes of nonconformance. The need for such actions will probably be identified by audits. Examples of this type of action include:
   - Staff training in technical skills or in implementing the QA Plan
   - Rescheduling laboratory routines to ensure analysis within allowed holding times
   - Identifying vendors to supply reagents of sufficient purity
   - Revision of the Contractor QA system or replacement of personnel

For either immediate or long-term corrective actions, steps comprising a closed-loop corrective action system are as follows:

- Define the problem
- Assign responsibility for investigating the problem
- Investigate and determine the cause of the problem
- Determine a corrective action to eliminate the problem
- Assign and accept responsibility for implementing the corrective action
- Establish effectiveness of the corrective action and implement the correction

- Verify that the corrective action has eliminated the problem

Depending on the nature of the problem, the corrective action employed may be formal or informal. In either case, occurrence of the problem, corrective action employed, and verification that the problem has been eliminated must be documented.

In addition, if the corrective action results in the preparation of a new standard or calibration solution(s), then a comparison of the new solution versus the old solution needs to be performed and the results supplied with the weekly QC submittal as verification that the problem has been eliminated.

Corrective action will be implemented depending on the scope of the action required and the type of control being enforced. The following sequence of steps should be followed by laboratory personnel or management when an out-of-control situation becomes apparent. If a problem is identified that threatens analytical results then analyses should be discontinued until the analytical sequence is demonstrated to be reliably stabilized. When a problem has been identified, the HLA QAC should be contacted and informed of the possible net affect of the problem on reported results. This is especially important when errors in calculations or improper processing are involved and the net affect upon previously analyzed samples may be unknown. In all cases when QC criteria are not achieved, affected samples should be reanalyzed, whenever possible, within the specified holding time.

The LQAC is responsible for documenting the corrective action performed during a project. A standard form will be used and contain all critical dates and information needed to trace the analytical results that may have been affected by any such action (see Table 11.1). All corrective action will be coordinated among the LQAC, the Laboratory Analytical Task Manager, and the appropriate analytical section supervisors. When a problem has been identified the complete analytical sequence should be reviewed to evaluate the true source of the problem. For example, all data processing procedures, calculations, blank results, calibrations, tuning parameters, interference checks, overall instrument sensitivity, logbook entries, standards or sample prepara-

Table 11.1: Corrective Action Record

Problem Analyte(s) _____ PMRMA I.D. No. _____

QUALITY ASSURANCE/QUALITY CONTROL
CORRECTIVE ACTION RECORD
(LABORATORY'S NAME)

_____

Quality Assurance Comments:

LQAC: _____ Date: _____

_____

Management Action:

Signature: _____ Date: _____

_____

Analyst Response (use back of form if needed):

Signature: _____ Date: _____

_____

Quality Assurance Approval:

QAC: _____ Date: _____

tion, chromatograms, quantification reports, and digestions should be checked and noted in the corrective action report. All corrective action will in this way document for later investigators the circumstances surrounding a specific type of analytical problem. Corrective action reports should be selectively filled out for each project and included in the final evidence file.

11.2 FIELD CORRECTIVE ACTION

During the initiation of field activities, the QAC will identify and enforce corrective action immediately onsite. This would include correction of sampling procedures that violate sample integrity, resampling of affected samples, repackaging of samples for shipment, and recompletion of COC forms or field measurement forms. Samples involved with corrective action performance will be documented along with the nature and extent of the required action. All correction activities will be recorded in the project field notebook and a copy of the corrective action note provided to the FQAC and Project Manager.

12.0 QUALITY CONTROL REPORTS

12.1 LABORATORY QC REPORTS

Normal submissions to PMRMA will include the Precertification and Certification Performance Data Packages, IRDMS submissions, audit reports, and the results of QC activities. During those periods when analyses are being conducted, all QC charts (tabular and graphical) will be submitted to the PMRMA Project QA Officer on a weekly basis. The QC report will be provided to the Project Officer not later than five working days after analyses for a week are completed. Analysis date will be defined by the day the analytical instrument was run. All points that indicate an out-of-control situation will be evaluated and explained. Any corrective measures and reanalysis of samples will be fully explained and documented, including procedural changes, to prevent recurrence. Printouts generated from control chart software programs provided by PMRMA will be utilized, when available. A checklist for inclusion with each control chart submission is shown in Table 12.1.

As an appendix to the project final report, the QAC, in conjunction with the LQAC and the Project Manager, will provide tabulation of all QC sample data, as well as specific observations delineating the control effectiveness for each analytical method. These observations will include the following:

- QC samples in each lot and how analytical results were combined to prepare control charts
- Spike levels and rationale for choosing those levels
- Possible effects on environmental sample results of detected concentrations in method blanks
- Unique matrix characteristics of environmental samples

If at any time during the analytical effort a process was not in control, a discussion will be submitted on:

- Rationale for judging a point as in control, if it appears to satisfy an out-of-control criterion
- Investigation of the out-of-control situation

Table 12.1: Control Chart Checklist
(One with Each Weekly Submission)

Contract/Task No. _____ Installation: _____

1. The following items are included in this weekly control chart package covering method(s) _____

2. _____ Summary

3. _____ X - R Control Charts for duplicate, high concentraton spiked QC samples, and Outlier Tests.

4. _____ X - R Three-point Moving Average Control Charts for low concentration spiked QC samples (Class 1), surrogate spiked standard matrix samples (Class 1A), Class 1B, extended range certifications (Class 1, Class 1A, and Class 1B), and Outlier Tests.

5. _____ Observations on each chart (when applicable).

   a. _____ Trend analysis

   b. _____ Out-of-control analysis

   c. _____ Actions taken

   d. _____ Demonstration of resumption of control

6. Recommendations

_____ _____
Contractor QAC Date

- Actions taken to bring the process back into control
- Actions taken to ensure that the out-of-control situation did not recur
- Disposition of data acquired while the process was out-of-control

The control chart checklist should be completed as described below:

Item 1 The PMRMA Method Number(s) under which the control charts were generated and that are included in this current package are to be listed in numerical order.

Item 2 A summary table will be prepared listing the method number(s), RMA lots, dates of analysis, and analytes that are included in this package.

Item 3 All X-bar and R Control charts generated in the analyses performed during this period will be included.

Items 3 and 4 Each control chart will include the following information:

- Analyte
- Method number
- Laboratory
- Spike concentration
- Chart title - one of the following:

  Single Day X-bar Control Chart
  Single Day R Control Chart
  Three-Point Moving Average X-bar Control Chart
  Three-Point Moving Average R Control Chart

- Three-letter lot designation and analysis date for each point, shown on the x-axis
- Percent Recovery (for X-bar control charts) or Range (for R control charts) along the y-axis
- Upper control limit (UCL), on X-bar and R control charts
- Mean, on X-bar and R control charts
- Lower warning limit (LWL), on X-bar control charts
- Lower control limit (LCL), on X-bar control charts

The charts must contain sufficient data so that any trends, if present, can be discerned. (Charts developed during the initial stages of analysis will contain all points. Charts developed after the process has been stabilized, at least 220 points,

will contain at a minimum the most recent 10 points). Any point(s) that exceeds the control limits will be flagged (by circling in red) for discussion under 5b below. Any Outlier Tests must be included.

Item 5 The observations made during the review of the control charts, including but not limited to the items listed, will be submitted in writing.

Item 5b An analysis of any points flagged on the control chart(s) as being out-of-control will be included. Discussion should attempt to describe the cause of the out-of-control status and whether the point(s) are to be expected due to the random statistics used to demonstrate control or are the results of a possible systematic error or bias that would affect the analytical results. The discussion should include evaluation of Outlier Test results.

Item 5c Describe all actions taken to get process back into control.

Item 5d The data generated to prove that the analysis is back in control along with the criteria used ascertaining same will be included.

Item 6 Recommendations made as to the acceptance or rejection of the lot analysis, based on Item 5 above.

12.2 FIELD QC REPORTS

Field QC Reports will be prepared and maintained in the project files. The review of field activities will include items presented in Appendix F. Field QC reports will provide a detailed review of site conditions experienced during the project. The emphasis of these reports will be on the identification of field related activities that may have affected physical or chemical results.

13.0 BIBLIOGRAPHY

American Society for Testing and Materials, 1973, Biological Methods for the Assessment of Water Quality, Special Technical Publication (STP) 528, J.C. Cairns and K.L. Dickson, editors, Philadelphia, Pennsylvania.

Bishop, T.A., A Statistical View of Detection and Determination Limits in Chemical Analyses, prepared for the Committee on Applications of Statistical Techniques in Chemical Problems, Battelle Columbus Laboratories, Columbus, Ohio.

Blank, L.T., 1980, Statistical Procedures for Engineering, management, and Science, McGraw-Hill Book Company, New York, New York.

Currie, L., 1968, Limits for Qualitative Detection and Quantitative Determination, Analytical Chemistry, Vol. 40, No. 3, pp. 586-593.

Federal Register, Part III, December 3, 1979, Guidelines Establishing Test Procedures for the Analysis of Pollutants, Vol. 44, No. 233, pp. 69464-69475.

Federal Register, Part III, November 29, 1983, Toxic Substances Control; Good Laboratory Practice Standards, Vol. 48, No. 230, pp. 53922-53944.

Federal Register, Part VIII, October 26, 1984, Guidelines Establishing Test Procedures for Analysis of Pollutants Under the Clean Water Act, Vol. 49, No. 209, pp. 43234-43442.

Huibaux, A., and Vos, G., 1970, Decision and Detection Limits for Linear Calibration Curves, Analytical Chemistry, Vol. 42, No. 8, pp. 849-855.

Kirchmer, C.J., 1983, Quality Control in Water Analyses, Environmental Science and Technology, Vol. 17, No. 4, pp. 174A-181A.

Kratochvil, B., and Taylor, J.K., 1981, Sampling for Chemical Analysis, Analytical Chemistry, Vol. 53, No. 8, pp. 924A-938A.

Natrella, M.G., 1963, Experimental Statistics NBS Handbook 91, National Bureau of Standards, Washington, D.C.

Program Manager Rocky for Mountain Arsenal Chemical Quality Assurance Plan, August 1989.

Scheffe, H., 1959, The Analysis of Variance, John Wiley and Sons, Inc., New York, New York, pp. 426-427.

Snedecor, G.W., and Cochran, W.G., 1980, Statistical Methods, 7th Edition, Iowa State University Press, Ames, Iowa.

Taylor, J.K., 1981, Quality Assurance of Chemical Measurements, Analytical Chemistry, Vol. 53, No. 14, pp. 1588A-1596A.

USATHAMA, 1987, Geotechnical Requirements for Drilling, Monitor Wells, Data Acquisition, and Reports, Aberdeen Proving Ground, Maryland.

U.S. Environmental Protection Agency, 1987a, A compendium of Superfund field operation methods: EPA/540/P-87/001, Office of Emergency and Remedial Response, Washington, D.C.

U.S. Environmental Protection Agency, 1987b, Data quality objectives for remedial response activities (example scenario - RI/FS activities at a site with contaminated soils and ground water).

U.S. Environmental Protection Agency, March 1987c, Data quality objectives for remedial response activities (development process): EPA/540/G-87/003, Office of Emergency and Remedial Response, Washington, D.C.

U.S. Environmental Protection Agency, 1986d, Data Quality objectives for the RI/FS.

U.S. Environmental Protection Agency, 1986l, Test methods for evaluating solid waste: Office of Solid Waste and Emergency Response (OSWER) Directive SW-846, Vol. 1B.

U.S. Environmental Protection Agency, April 1985g, Guidance for remedial investigations under CERCLA.

U.S. Environmental Protection Agency, 1985, Guidance on feasibility studies under CERCLA: Hazardous Waste Engineering Research Laboratory, Office of Research and Development, Cincinnati, Ohio.

U.S. Environmental Protection Agency, 1983a, Handbook for evaluating remedial action technology plans: Arthur D. Little, Inc.

U.S. Environmental Protection Agency, October 1983b, Standard operating procedures for the ICP-DES determination of trace elements in suspected particulate matter collected on glass-fiber filters: Environmental Monitoring Systems Laboratory, prepublication copy.

U.S. Environmental Protection Agency, 1982a, Handbook: remedial action at waste disposal sites, technology transfer: EPA/625/6-82/006, Office of Emergency and Remedial Response, Washington, D.C.

U.S. Environmental Protection Agency, 1980a, Extraction and analysis of priority pollutants in biological tissue: S&A Division, Region IV, Laboratory Services Branch, Athens, Georgia.

U.S. Environmental Protection Agency, 1980b, Interim methods for the sampling and analysis of priority pollutants in sediments and fish tissue: ESML, Cincinnati, Ohio.

U.S. Environmental Protection Agency, 1980d, Management of Hazardous Waste Leachate: SW-871, Touhill, Shuckrow and Associates.

U.S. Environmental Protection Agency, 1980e, Treatability manual, vol I, treatability data, and treatability manual, vol. III, technologies for control removal of pollutants: Office of Emergency and Remedial Response, EPA/600/8-80/042 A and C.

U.S. Environmental Protection Agency, 1973, Biological Field and Laboratory Methods for Measuring the Quality of Surface Waters and Effluents, EPA-670/4-73-001, Environmental Research Center, Cincinnati, Ohio.

U.S. Environmental Protection Agecny, 1976, Quality Assurance Handbook for Air Pollution Measurement Systems: Volume I Principles, EPA-600/9-76-005, Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina.

U.S. Environmental Protection Agency, 1977a, NPDES Compliance Sampling Manual, Office of Water Enforcement, Washington, D.C.

U.S. Environmental Protection Agency, 1977b, Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II - Ambient Air Specific Methods, Environmental Protection Agency-600/4-77-027a, Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina.

U.S. Environmental Protection Agency, 1977c, Quality Assurance Handbook for Air Pollution Measurement Systems: Volume III - Stationary Source Specific Methods, Environmental Protection Agency-600/4-77-027b, Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina.

U.S. Environmental Protection Agency, 1979a, Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA-600/4-79-019, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

U.S. Environmental Protection Agency, 1979b, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

U.S. Environmental Protection Agency, 1980a, Guidelines and Specifications for Preparing Quality Assurance Program Plans, QAMS-004/80, Office of Monitoring Systems and Quality Assurance, Washington, D.C.

U.S. Environmental Protection Agency, 1980b, Guidelines and specifications for Preparing Quality Assurance Project Plans, MERL/QA-2, Municipal Environmental Research Laboratory, Cincinnati, Ohio.

U.S. Environmental Protection Agency, 1980c, Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80, Office of Monitoring Systems and Quality Assurance, Washington, D.C.

U.S. Environmental Protection Agency, 1980d, Samplers and Sampling Procedures for Hazardous Waste Streams, EPA-600/2-80-018, Municipal Environmental Research Laboratory, Cincinnati, Ohio.

U.S. Environmental Protection Agency, 1982a, Handbook for Sampling and Sample Preservation of Water and Wastewater, EPA-600/4-82-029, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

U.S. Environmental Protection Agency, 1982b, NEIC Policies and Procedures, EPA-330/9-78-001-R, National Enforcement Investigations Center, Denver, Colorado.

U.S. Environmental Protection Agency, 1982c, Quality Assurance Manual for the Environmental Sciences Research Laboratory, ESQA-002/82, Environmental Sciences Research Laboratory, Research Triangle Park, North Carolina.

U.S. Environmental Protection Agency, 1982d, Technical Enforcement Guidance: Groundwater Monitoring Assessment Programs at Interim Status Facilities, SW-954, Office of Solid Waste and emergency Response, Washington, D.C.

U.S. Environmental Protection Agency, 1982e, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, Second Edition, Office of Solid Waste and Emergency Response, Washington, D.C.

U.S. Environmental Protection Agency, 1982f, Test Methods: Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, EPA-600/4-82-057, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

U.S. Environmental Protection Agency, 1982g, Test Methods: Technical Additions to Methods for Chemical Analysis of Water and Wastes, EPA-600/4-82-055, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

U.S. Environmental Protection Agency, 1983, Calculation of Data Quality Indicators, Office of Research and Development, Washington, D.C.

U.S. Environmental Protection Agency, 1983b, Preparation of Soil Sampling Protocol: Techniques and Strategies, EPA-600/4-83-020, Environmental Monitoring Systems Laboratory, Las Vegas, Nevada.

U.S. Environmental Protection Agency, 1983c, Quality Assurance Handbook for Air Pollution Measurement Systems: Volume IV Meteorological Measurements, Research Triangle Park, North Carolina.

U.S. Environmental Protection Agency, 1983d, Quality Assurance Handbook for Air Pollution Measurement Systems: Volume V - Manual for Precipitation Measurement Systems, EPA 600/4-82-042a and b, Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina.

U.S. Environmental Protection Agency, 1984e, Characterization of Hazardous Waste Sites - A Methods Manual: Volume II - Available Sampling Methods, Second Edition, EPA 600/4-84-076, Environmental Monitoring Systems Laboratory, Las Vegas, Nevada.

U.S. Environmental Protection Agency, 1984b, Remedial Investigations Guidance Document. Office of Emergency and Remedial Response, Washington, D.C.

U.S. Environmental Protection Agency, 1984c, Soil Sampling Quality Assurance User's Guide, EPA 600/4-84-043, Environmental Monitoring Systems Laboratory, Las Vegas, Nevada.

U.S. Geological Survey, 1977a, Techniques of Water-Resource Investigations of the United States Geological Survey: Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples, Book 5, Chapter A4, U.S. Department of the Interior, Washington, D.C.

U.S. Geological Survey, 1977b and subsequent revisions, National handbook of Recommended Methods for Water-Data Acquisition, Office of Water Data Coordination, Reston, Virginia.

Weston, A.G., 1984, Obtaining Reliable Priority - Pollutant Analyses, Chemical Engineering, Vol. 91, No. 9, pp. 54-60.

Wilson, A.L., 1970, The Performance Characteristics of Analytical Methods - I and II, Talanta, Vol. 17, pp. 21-44.

Youden, W.J., 1964, Statistical Methods for Chemists, John Wiley and Sons, New York, New York.

Appendix A

FORMAT FOR DOCUMENTATION OF ANALYTICAL METHODS

FORMAT FOR DOCUMENTATION OF ANALYTICAL METHODS PRECERTIFICATION CALIBRATION

TITLE

I. SUMMARY

A. ANALYTE(S). State analyte(s) that can be analyzed by this method (e.g., 246TNT).

B. MATRIX. Environmental matrix (or matrices) for which the method is applicable (e.g., ground water and surface water).

C. GENERAL METHOD. Brief method description (e.g., direct injection HPLC with UV detection).

II. APPLICATION

A. CALIBRATION RANGE. Concentration range of the calibration standards (representative of the extract).

B. TESTED CONCENTRATION RANGE. Based upon the procedure, the calculated equivalent tested concentration range in the original matrix (e.g., 1 - 20 ug/L in water).

C. SENSITIVITY. Instrumental response observed for absolute quantity of analyte at the calculated reporting limit (e.g., 1500 area units for 40 picograms).

D. INTERFERENCES. Any observed interferences or any interferences anticipated based on the method of analysis.

E. SAFETY INFORMATION. Special health hazards and safety precautions for handling samples, solutions, and chemicals.

III. APPARATUS AND CHEMICALS

A. INSTRUMENTATION. Makes and models of instruments and associated components. Operating parameters of instruments and associated components. Retention times and retention time windows.

B ANALYTES. Chemical Abstracts Service registry number and basic physical properties.

C. REAGENTS AND STANDARD ANALYTICAL REFERENCE MATERIALS. Identity, concentration, purity, and source of reagents and SARMs.

IV. PRECERTIFICATION CALIBRATION

A. Preparation of Standards. Step-by-step preparation procedures, including proper storage, shelf-life, and concentrations.

B. Instrument Calibration. Detailed procedures for instrument tuning and analysis of standards.

C. Analysis of Calibration Data. Criteria for acceptability.

V. PROCEDURE

A. SEPARATIONS.

B. CHEMICAL REACTIONS.

C. INSTRUMENTAL ANALYSIS.

VI. CALCULATIONS. Detailed procedure for calculating sample concentrations from the instrument responses, including calibration curves and formulae.

VII. REFERENCES. Published references for the procedures described.

VIII. DATA

A. Response versus concentration data.

B. Response versus concentration graphs.

C. Lack Of Fit tests.

D. Zero Intercept tests.

FORMAT FOR DOCUMENTATION OF ANALYTICAL METHODS

PART II. CERTIFICATION CALIBRATION

I. SUMMARY

A. Analyte(s): state analyte(s) that can be analyzed by this method (e.g., 2, 4, 6-TNT).

B. Matrix: environmental matrix (or matrices) for which the method is applicable (e.g., ground water and surface water).

C. General method: brief method description (e.g., direct injection HPLC with UV detection).

III. APPLICATION

A. Tested concentration range: tested concentration range in the original matrix (e.g., 1-20 ug/L in water).

B. Sensitivity: instrumental response observed for absolute quantity of analyte at the calculated reporting limit (e.g., 1500 area units for 40 picograms).

C. Reporting limit: certified reporting limit for complete analytical method determined from found versus actual concentrations for spiked standard matrix samples and calculated according to PMRMA reporting limit program, expressed in terms of concentration in original matrix.

D. Analysis rate: estimated maximum number of samples that can be analyzed by this method in an 8-hour day after instrument calibration.

F. Safety information: Special health hazards and safety precautions for handling samples, solutions, and chemicals.

III. APPARATUS AND CHEMICALS

A. Glassware/hardware: quantities and sizes of all miscellaneous equipment, including sources of specialty or trademark items.

B. Instrumentation: makes and models of instruments and associated components. Operating parameters of instruments and associated components. Retention times and retention windows (to include the criteria used in setting the retention time windows).

C. Analytes; chemical abstracts service registry number and basic physical properties.

D. Reagents and SARMS: identity, concentration, purity, and source of reagents and SARMS.

IV. CALIBRATION

A. Initial Calibration

1. Preparation of standards: step-by-step preparation procedures, including proper storage, shelf-life, and concentrations.

2. Instrument calibration: detailed procedures for instrument tuning and analysis of standards.

3. Analysis of calibration data: criteria for acceptability, based on precertification calibration curve.

B. Daily calibration

1. Preparation of standards: step-by-step preparation procedures, including proper storage, shelf-life, and concentrations.

2. Instrument calibration: detailed procedures for instrument tuning and analysis of standards.

3. Analysis of calibration data: criteria for acceptability.

4. Calibration checks: as used internally by laboratory and/or specified by this QA Plan.

V. CERTIFICATION TESTING. Control spikes. Step-by-step procedures for preparing standard matrix certification samples.

VI. SAMPLE HANDLING AND STORAGE

A. Sampling procedure: special considerations due to the nature of the analyte; required preservation procedures.

B. Containers.

C. Storage conditions: conditions required in the field and the laboratory to maintain sample integrity.

D. Holding time limits.

E. Solution verification: description of measures to verify integrity of working calibration and control spike solutions.

VII. PROCEDURE

A. Separations

B. Chemical reactions

C. Instrument analysis

VIII. CALCULATIONS. Detailed procedure for calculating sample concentrations from the instrument responses, including calibration curves and formulae.

IX. DAILY QUALITY CONTROL

A. Control samples: detailed, step-by-step procedure for preparing spiked QC samples, including spiking concentrations based on Certified Reporting Limit.

B. Control charts: description of charts to be maintained. List initial warning and control limits as obtained from certification.

X. REFERENCES. Published references for the procedures described.

XI. DATA

A. Off-the-shelf analytical reference materials characterization.

B. Initial Calibration

1. Response versus concentration data.

2. Response versus concentration graphs.

C. Daily Calibration

1. Response.

2. Required percentage or two standard deviation limits.

D. Standard Certification Samples.

1. Tabulation and graph of found versus target concentrations.

2. Lack of Fit and Zero Intercept tests for the pooled data.

3. Calculated lease squares, linear regression line, confidence bounds, reporting limit, accuracy, standard deviation, percent imprecision, and percent inaccuracy.

4. Chromatograms from each day of certification analyses for the highest tested concentration and for the tested concentration closest to the calculated reporting limit. Each chromatogram shall be labeled with the analysis data, analysis time, target concentration, test name, and reference to the laboratory logbook where analytical activities were recorded. The identity of each peak shall also abe labeled.

DOCUMENTATION FOR NON-CERTIFIED METHODS

1. Organization submitting documentation
2. Statement of analysis to be performed
3. Description of the EPA or ASTM standard method technical approach to be followed
4. Specific details of instrumentation to be used, solvents, calibration, and internal QA/QC procedures
5. Laboratory qualifications and resources required to perform the uncertified method or technique
6. Calculations or detailed procedures for estimating sample characteristic or concentration from instrument response
7. Calibration curves and formulae if applicable to the type of uncertified method employed

DOCUMENTATION FOR PROPOSED METHOD DEVELOPMENT

1. Organization submitting documentation
2. Statement of the problem
3. Description of the technical approach to be followed
4. Specific details on procedures, solvents, instrumentation, etc.
5. Estimate of resources required to include labor hours, funds, and schedule

Appendix B

SAMPLE OUTPUT OF PMRMA STATISTICAL ANALYSIS PROGRAM REQUIRED FOR CERTIFICATION

SAMPLE OUTPUT OF PMRMA STATISTICAL ANALYSIS
PROGRAM REQUIRED FOR CERTIFICATION

PRE-CERTIFICATION ANALYSIS

Report Date: 04/09/87
Page: 1
Laboratory: QQ
Analysis Date: 12/01/85
Matrix: WA

Method Name: EXAMPLE
Compound: TEST
Units of Measure: UGL

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept ---
Y = (3.734532290) + (1001.336550)X

- Model through the Origin -
Y - (1003.447870)X

	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	164.9304260	8	20.61630325	235.6420900	9	26.18245444
Total Error:	49.50000000	5	9.900000000	49.50000000	5	9.900000000
Lack of Fit:	115.4304260	3	38.47680867	186.1420900	4	46.53552250

LOF F-Ratio (F): 3.886546330 LOF F-Ratio(F): 4.700557828
Critical 95% F: 5.41 Critical 95% F: 5.19

ZERO INTERCEPT HYPOTHESIS

Zero Intercept Accepted

Calculated F: 3.429890565 Critical 95% F: 5.32

****

TABLE OF DATA POINTS

Targets: 5 Measures per Target: 2

	Target Value	Instrument Values	
1:	0.1000000	102	99
2:	0.2200000	223	225
3:	0.4400000	441	447
4:	1.1000000	1109	1114
5:	2.5000000	2502	2507

*** END OF PRE-CERTIFICATION DATA TABLE ***

CERTIFICATION ANALYSIS

Report Date: 04/09/98

Method Name: EXAMPLE
Compound: TEST
Units of Measure: UGL

Laboratory: QQ
Analysis Date: 12/01/85
Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept ---
Y = (0.037863471) + (0.897710724) X

- Model through the Origin -
Y = (0.922155819)X

	(SS)	(df)	(MS)	(SS)	(df)	(MS)
Residual:	0.078619297	18	0.004367739	0.092223765	19	0.004853882
Total Error:	0.071763000	15	0.004784200	0.071763000	15	0.004784200
Lack of Fit:	0.006856297	3	0.002285432	0.020460765	4	0.005115191

LOF F-Ratio(F): 0.477704158
Critical 95% F: 3.29

LOF F-Ratio(F): 1.069184263
Critical 95% F: 3.06

ZERO INTERCEPT HYPOTHESIS

Zero Intercept Accepted

Calculated F: 3.114762493 Critical 95% F: 4.41

****

TABLE OF DATA POINTS

Targets: 5

Measures per Target: 4

	Target Value	Found Concentration			
1:	0.1100000	0.1800000	0.1360000	0.1390000	0.1520000
2:	0.2200000	0.2800000	0.2460000	0.2480000	0.2390000
3:	0.4400000	0.4200000	0.3850000	0.3970000	0.4110000
4:	1.1000000	1.0300000	1.0300000	1.0670000	0.9200000
5:	2.2000000	2.0900000	1.8650000	2.1760000	1.9610000

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS

Report Date: 04/09/87

Method Name: EXAMPLE
Compound: TEST
Units of Measure: UGL

Laboratory: QQ
Analysis Date: 12/01/85
Matrix: WA

TABLE OF RESULTS FOR TRUNCATED DATA SET

Target Concentration	Standard Deviation	Percent Inaccuracy	Percent Imprecision
0.1100000	0.0200728	37.954545	13.227535
0.2200000	0.0182460	15.113636	7.2047400
0.4400000	0.0154137	-8.352273	3.8223778
1.1000000	0.0636049	-8.022727	6.2866222
2.2000000	0.1374854	-8.045455	6.7960724

CERTIFICATION ANALYSIS

Report Date: 04/09/87

Method Name: EXAMPLE
Compound: TEST
Units of Measure: UGL

Laboratory: QQ
Analysis Date: 12/01/85
Matrix: WA

TABLE OF DATA POINTS

Target Concentration	Found Concentration
0	0
	0
	0
	0
0.1100000	0.1800000
	0.1360000
	0.1390000
	0.1520000
0.2200000	0.2800000
	0.2460000
	0.2480000
	0.2390000
0.4400000	0.4200000
	0.3850000
	0.3970000
	0.4110000
1.1000000	1.0300000
	1.0300000
	1.0670000
	0.9200000
2.2000000	2.0900000
	1.8650000
	2.1760000
	1.9610000

CERTIFICATION ANALYSIS

Report Date: 04/09/87

Method Name: EXAMPLE
Compound: TEST
Units of Measure: UGL

Laboratory: QQ
Analysis Date: 12/01/85
Matrix: WA

--- REGRESSION EQUATION ---
Y = 0.09049594X + 0.0266359

--- UPPER REPORTING LIMIT ---
2.2000000

--- SLOPE ---
0.9049594

SUMMARY TRUNCATION TABLE

Target Concentrations Used	Slope	% Change from Total Data Set	% Change from Previous Data Set
Entire data set	0.904594	0	0
Minus 1 highest	0.8919376	1.4389388	1.4389388
Minus 2 highest	0.89019848	1.6315183	0.1953911

Target Concentrations Used	Certified Reporting Limit	Upper Reporting Limit
Entire data set	0.2410763	2.2000000
Minus 1 highest	0.1525508	2.2000000
Minus 2 highest	0.1300442	2.2000000

Appendix C

CONTAINERS, PRESERVATION, STORAGE, AND HOLDING TIMES

Appendix C
Containers, Preservation, Storage, and Holding Times[1]

Parameter	Container[2]		Preservative[3,4]		Maximum Holding Time for all Matrices[5]
	Water	Solids	Water	Solids	
INORGANIC TESTS					
Alkalinity	P	S	Cool, 4°C	Cool, 4°C	14 days
Ammonia	P	S	Cool, 4°C	Cool, 4°C	28 days
Bicarbonate	P	S	None Required	None Required	Analyze Immediately
Biochemical Oxygen Demand (BOD) abd /carbibaceiys BOD	P	S	Cool, 4°C	Cool, 4°C	48 hours
Bromide	P	S	None Required	None Required	28 days
Carbonate	P	S	None Required	None Required	Analyze Immediately
Chemical Oxygen Demand (COD)	P	S	Cool, 4°C H_2SO_4 to pH <2	Cool, 4°C	28 days
Chloride	P	S	None Required	None Required	28 days
Chlorine, Total Residual	P	N/A	None Required	N/A	Analyze Immediately
Dissolved Oxygen Probe	G	N/A	None Required	N/A	Analyze Immediately
Fluoride	P	S	None Required	None Required	28 days
Hardness	P	N/A	HNO_3 or H_2SO_4 to pH <2	N/A	6 months
Kjeldahl and Organic Nitrogen	P	S	Cool, 4°C H_2SO_4 to pH <2	Cool, 4°C	28 days
Anions	P	S	Cool, 4°C	Cool, 4°C	28 days
Metals[7]					
Chromium VI	P	S	Cool, 4°C	Cool, 4°C	24 hours
Mercury	P	S	HNO_3 to pH <2	Cool, 4°C	28 days
ICAP Metals	P	S	HNO_3 to pH <2	Cool, 4°C	6 months
Arsenic	P	S	HNO_3 to ph <2	Cool, 4°C	6 months

Appendix C (Continued)

Parameter	Container[2] Water	Container[2] Solids	Preservative[3,4] Water	Preservative[3,4] Solids	Maximum Holding Time for all Matrices[5]
INORGANIC TESTS (Continued)					
Nitrate	P	S	Cool, 4°C	Cool, 4°C	48 hours
Nitrate plus Nitrite	P	S	Cool, 4°C	Cool, 4°C	28 days
Nitrite	P	S	Cool, 4°C	Cool, 4°C	48 hours
Oil and Grease	G	S	Cool, 4°C H_2SO_4 to pH <2	Cool, 4°C	28 days
Orthophosphate	P	S	Filter Immediately Cool, 4°C	Cool, 4°C	48 hours
pH	P	S	None Required	None Required	Analyze Immediately
Phosphorous, Total	P,G	S	Cool, 4° H_2SO_4 to pH <2	Cool, 4°C	28 days
Residue					
Filterable	P	N/A	Cool, 4°C	N/A	7 days
Settleable	P	N/A	Cool, 4°C	N/A	48 hours
Nonfilterable (TSS)	P	N/A	Cool, 4°C	N/A	7 days
Total	P	N/A	Cool, 4°C	N/A	7 days
Volatile	P	N/A	Cool, 4°C	N/A	7 days
Specific Conductance	P	S	Cool, 4°C	Cool, 4°C	28 days
Sulfate	P	S	Cool, 4°C	Cool, 4°C	28 days
Sulfide	P	S	Cool, 4°C Add Zinc Acetate plus NaOH to pH >9	Cool, 4°C	7 days
Temperature	P	S	None Required	None Required	Analyze Immediately
Turbidity	P	N/A	Cool, 4°C	N/A	48 hours

Parameter	Container[2] Water	Container[2] Solids	Preservative[3,4] Water	Preservative[3,4] Solids	Maximum Holding Time for all Matrices[5]
ORGANIC TESTS[8]					
Volatile Organics (GC/MS)*	V	V	Cool, 4°C	Cool, 4°C	7 days
Semivolatile Organics (GC/MS)	V	S	Cool, 4°C	Cool, 4°C	7 days
Pesticides					
Organochlorine	G	S	Cool, 4°C	Cool, 4°C	7 days
Organosulfur	G	S	Cool, 4°C	Cool, 4°C	7 days
Organophosphorus	G	S	Cool, 4°C	Cool, 4°C	7 days
DBCP	G	S	Cool, 4°C	Cool, 4°C	7 days
Phosphonates	G	S	Cool, 4°C	Cool, 4°C	7 days
Hydrocarbons	V	S	Cool, 4°C	Cool, 4°C	7 days
Purgeable Aromatic Hydrocarbons	V	V	Cool, 4°C 0.008% $Na_2S_2O_3$[6] HCl to pH <2	Cool, 4°C	7 days
Purgeable Organohalogens	V	V	Cool, 4°C 0.008% $Na_2S_2O_3$[6]	Cool, 4°C	7 days
Total Organic Carbon	G	S	Cool, 4°C HCl or H_2SO_4 to pH <2	Cool, 4°C	28 days

Parameter	Container[2]	Preservative Method (Biota Only)	Maximum Holding Time (Biota Only)
Pesticides - fat, nonfat (GC/ECD)	E	Cool, 4°C	4 years**
Metals			
Mercury (AAS cold vapor)	E	Cool, 4°C	4 years**
Arsenic (GFAA)	E	Cool, 4°C	4 years**
Other Metals (ICP)	E	Cool, 4°C	4 years**

*Volatile organics (GC/MS) and purgeable aromatics should be preserved with HCL to pH <2 to achieve the holding time. Discussions are currently underway with USATHAMA's analytical branch to better define the preservation and holding time requirements for these analytes.

**After a biota sample is extracted/digested, the holding time is the same as that of a soil sample.
Analytes not listed should be preserved at 4°C and held not longer than 7 days.

[1]Preservatives and holding times are from Federal Register, Vol. 49, No. 209, Friday, October 26, 1984, Page 43260 and Characaterization of Hazardous Waste Sites: A Methods Manual -- Volume II, Sampling Methods, Second Edition, EPA-600/4-84-076. Container requirements are consistent with these references.

[2]P = Polyethylene
E = Ziplock plastic bag
G = 1-Liter amber glass with Teflon-lined cap
V = 2-40 ml amber glass with Teflon septum
S = 8-oz amber glass wide-mouth jar with Teflon-lined cap
o = 2-25 ml amber glass with Teflon-lined cap

[3]Sample preservation should be performed immediately upon sample collection. For composite samples, each aliquot should be preserved at the time of collection. When use of an automatic sampler makes it impossible to preserve each aliquot, samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

[4]When any sample is to be shipped by common carrier or sent through the U.S. Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements in this table, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation, has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by wieght or less (pH about 12.3 or less).

[5]Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Some samples may not be suitable for the maximum time period given in the table. A laboratory is obligated to hold the sample for a shorter time if knowledge exists to show this is necessary to maintain sample integrity.

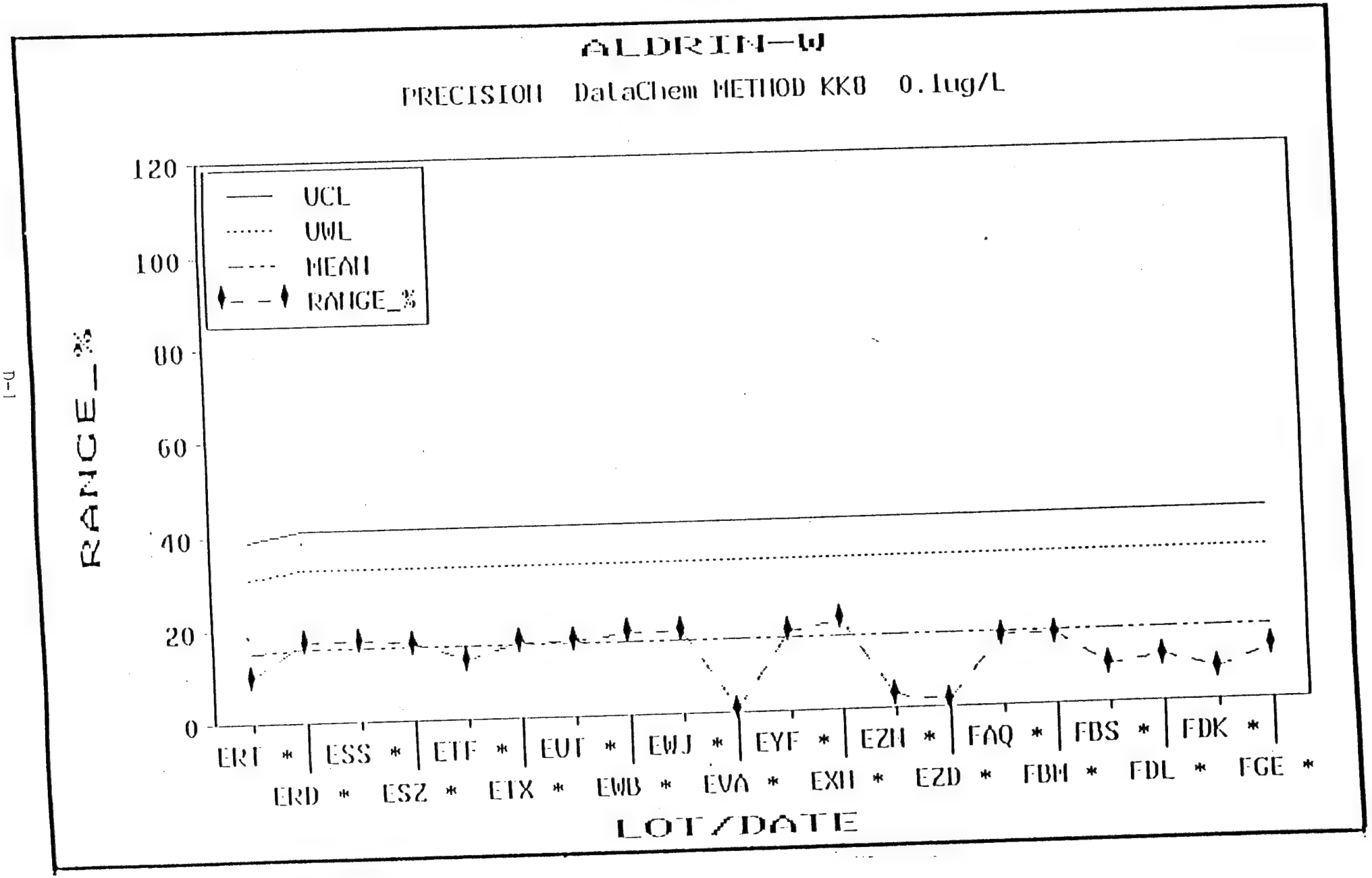
[6]Should only be used in the presence of residual chlorine.

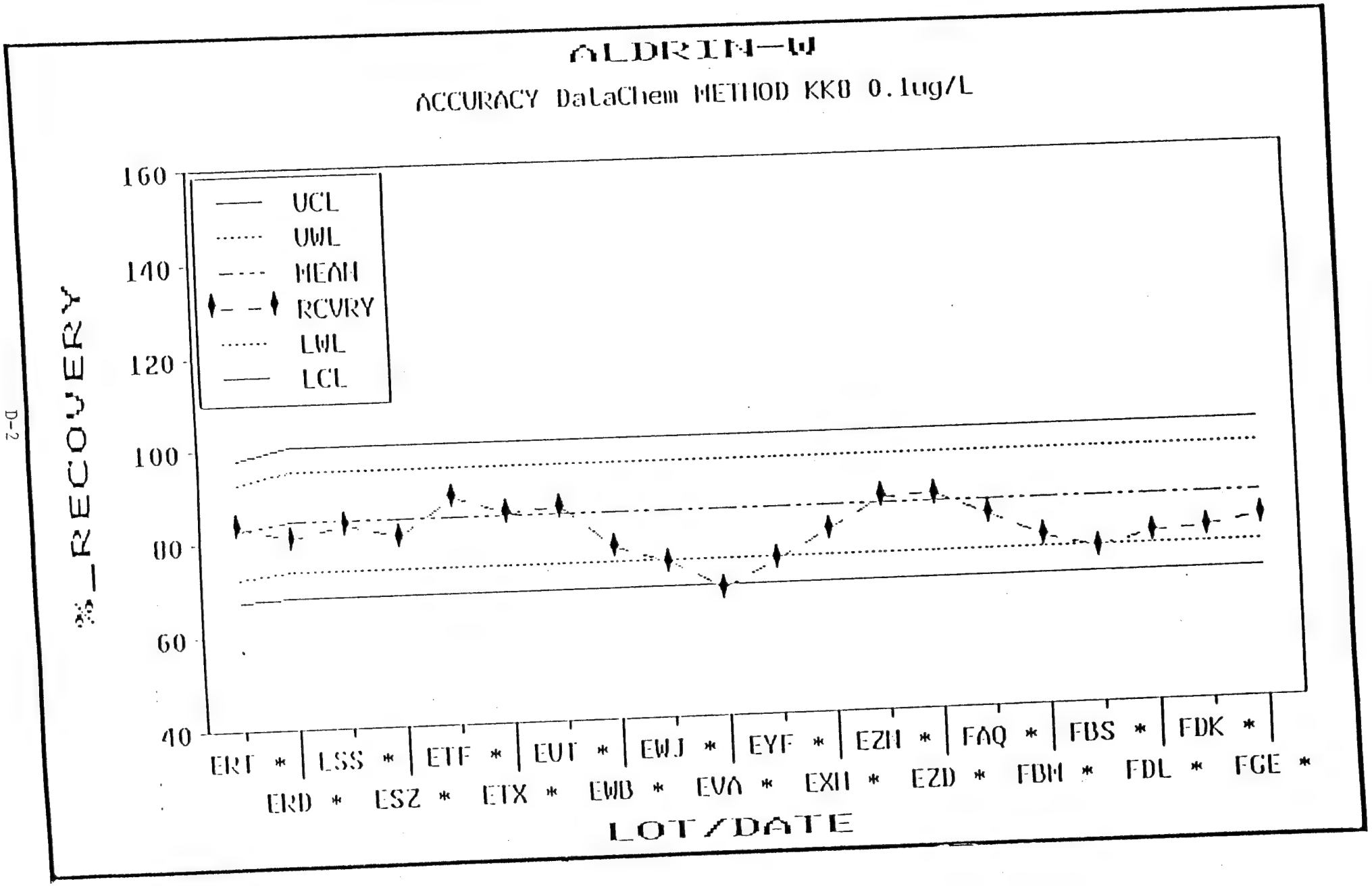
[7]For dissolved metals, filter immediately onsite before adding preservative.

[8]Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

Appendix D

CONTROL CHART FOR CLASS 1 CERTIFIED METHOD


ALDRIN-W
PRECISION DataChem METHOD KK8 0.1ug/L
UCL
UWL
MEAN
RANGE_%
120
100
80
60
40
20
0
RANGE_%
ERT * ESS * ETF * EUT * EWJ * EYF * EZH * FAQ * FBS * FDK *
ERD * ESZ * ETX * EWB * EVA * EXH * EZD * FBH * FDL * FGE *
LOT/DATE


ALDRIN-W
ACCURACY DaLaChem METHOD KK0 0.1ug/L
UCL
UWL
MEAN
RCVRY
LWL
LCL
160
140
120
100
80
60
40
%_RECOVERY
ERT * LSS * ETF * EUT * EWJ * EYF * EZH * FAQ * FBS * FDK *
ERD * ESZ * ETX * EWB * EVA * EXH * EZD * FBM * FDL * FGE *
LOT/DATE

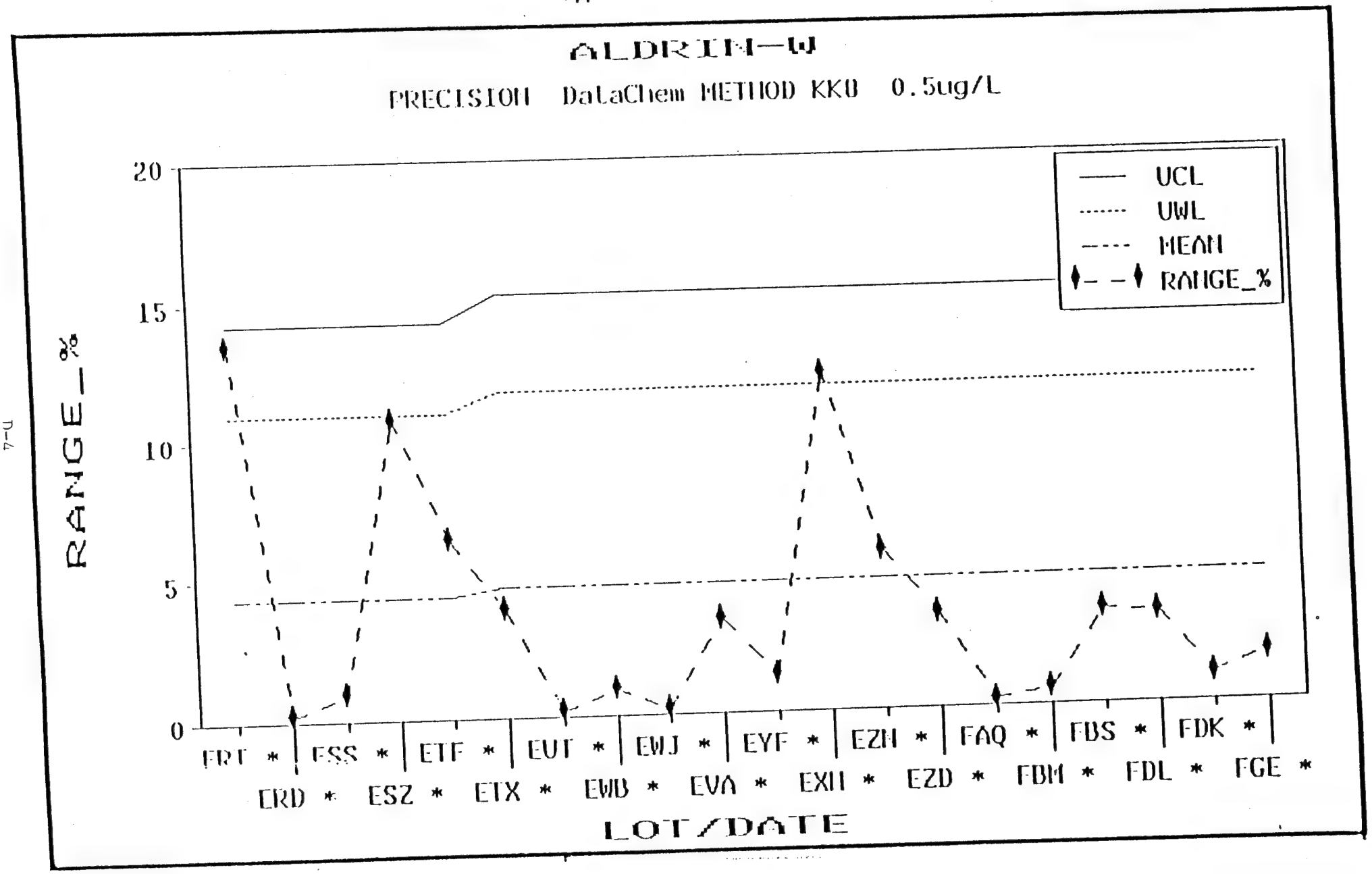
21-DEC-1988 Page: 1

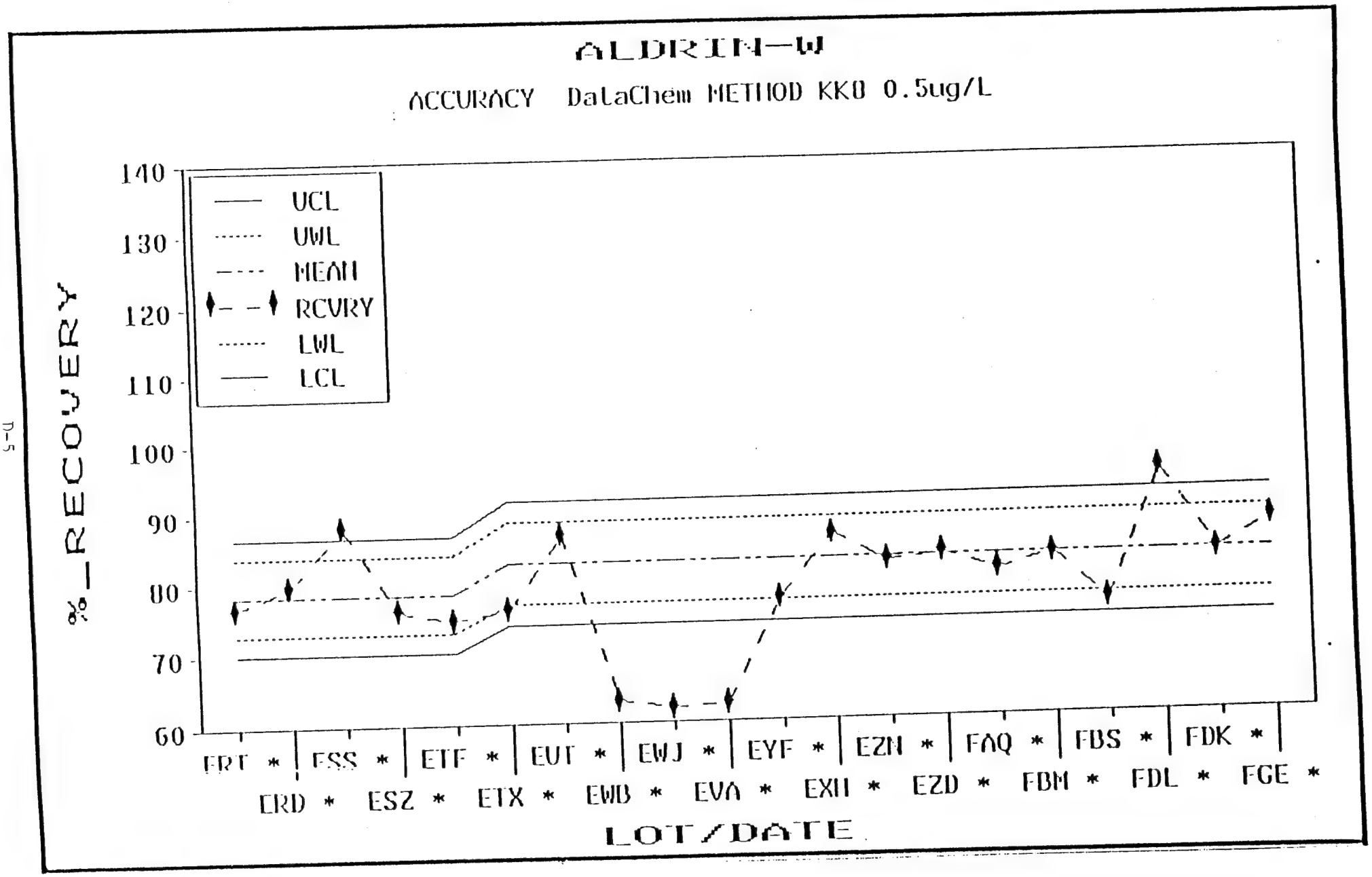
ALDRIN-W

LOW SPIKE VALUE

REC #	LOT	DATE	TARGET	FOUND	RECOVERY	MEAN RECOVERY
101	ERT	28-OCT-1988	0.100	0.0893	89.30	83.37
102	ERD	29-OCT-1988	0.100	0.0720	72.00	80.87
103	ESS	31-OCT-1988	0.100	0.0887	88.70	83.33
104	ESZ	3-NOV-1988	0.100	0.0818	81.80	80.83
105	ETF	3-NOV-1988	0.100	0.0947	94.70	88.40
106	ETX	5-NOV-1988	0.100	0.0783	78.30	84.93
107	EUT	7-NOV-1988	0.100	0.0848	84.80	85.93
108	EWB	10-NOV-1988	0.100	0.0670	67.00	76.70
109	EWJ	14-NOV-1988	0.100	0.0678	67.80	73.20
110	EVA	15-NOV-1988	0.100	0.0671	67.10	67.30
111	EYF	17-NOV-1988	0.100	0.0840	84.00	72.97
112	EXH	18-NOV-1988	0.100	0.0864	86.40	79.17
113	EZH	21-NOV-1988	0.100	0.0868	86.80	85.73
114	EZD	22-NOV-1988	0.100	0.0851	85.10	86.10
115	FAQ	30-NOV-1988	0.100	0.0718	71.80	81.23
116	FBH	30-NOV-1988	0.100	0.0701	70.10	75.67
117	FBS	5-DEC-1988	0.100	0.0780	78.00	73.30
118	FDL	6-DEC-1988	0.100	0.0801	80.10	76.07
119	FDK	12-DEC-1988	0.100	0.0730	73.00	77.03
120	FGE	12-DEC-1988	0.100	0.0840	84.00	79.03

MEAN RECOVERY:	84.30
ACCURACY UPPER CONTROL LIMIT:	100.62
ACCURACY LOWER CONTROL LIMIT:	67.98
AVERAGE RANGE:	15.95
PERCISION CONTROL LIMIT:	41.08


ALDRIN-U
PRECISION DataChem METHOD KKU 0.5ug/L
UCL
UWL
MEAN
RANGE_%
RANGE_%
20
15
10
5
0
FRT * FSS * ETF * EUT * EWJ * EYF * EZH * FAQ * FBS * FDK *
ERD * ESZ * ETX * EWB * EVA * EXH * EZD * FBM * FDL * FGE *
LOT/DATE


ALDRIN-W
ACCURACY DataChem METHOD KK0 0.5ug/L
UCL
UWL
MEAN
RCVRY
LWL
LCL
140
130
120
110
100
90
80
70
60
%_RECOVERY
FRT * FSS * ETF * EUT * EWJ * EYF * EZN * FAQ * FBS * FDK *
ERD * ESZ * ETX * EWB * EVA * EXH * EZD * FBM * FDL * FGE *
LOT/DATE

20-DEC-1988 Page: 1

ALDRIN-W

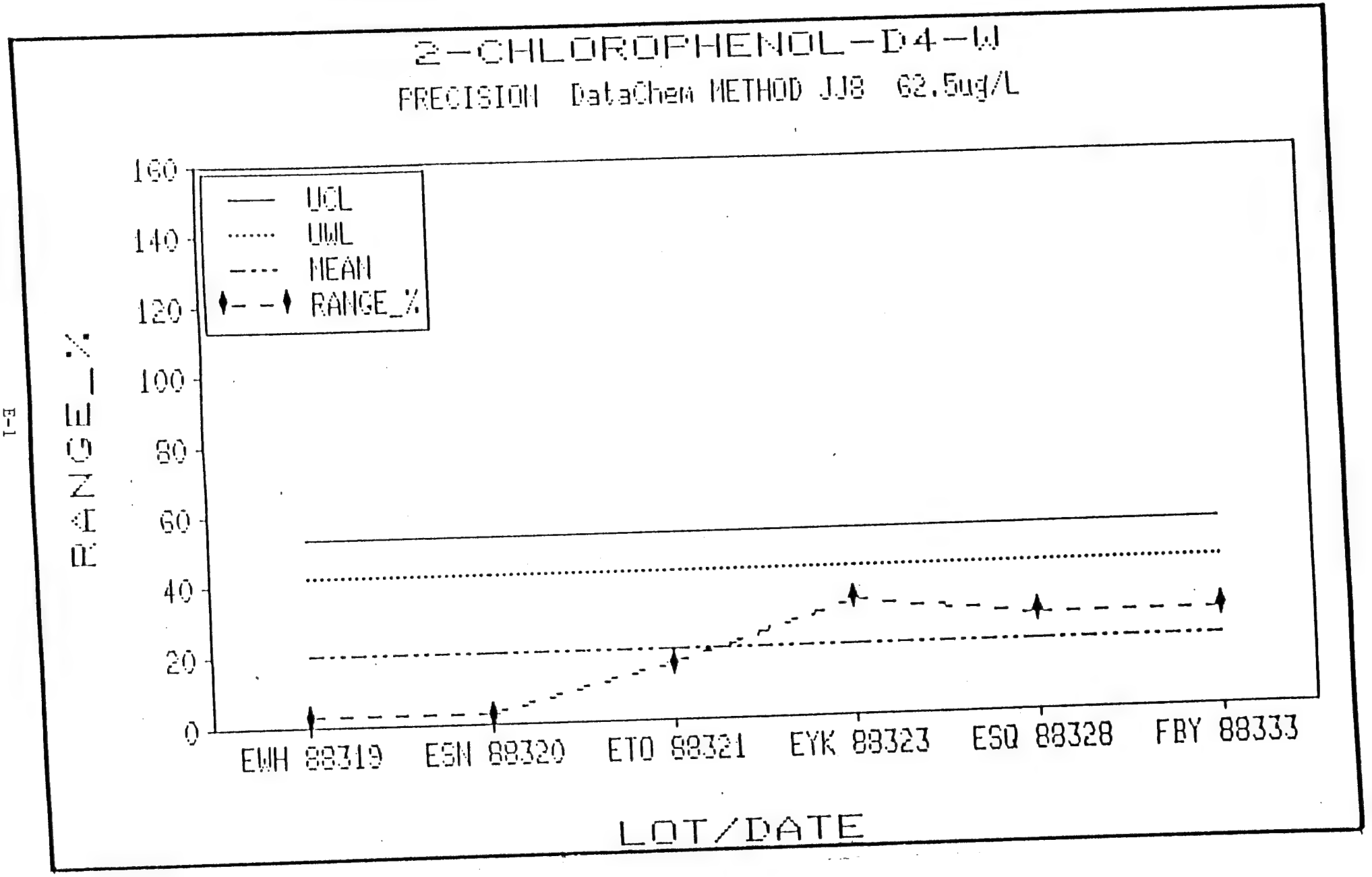
HIGH SPIKE VALUE

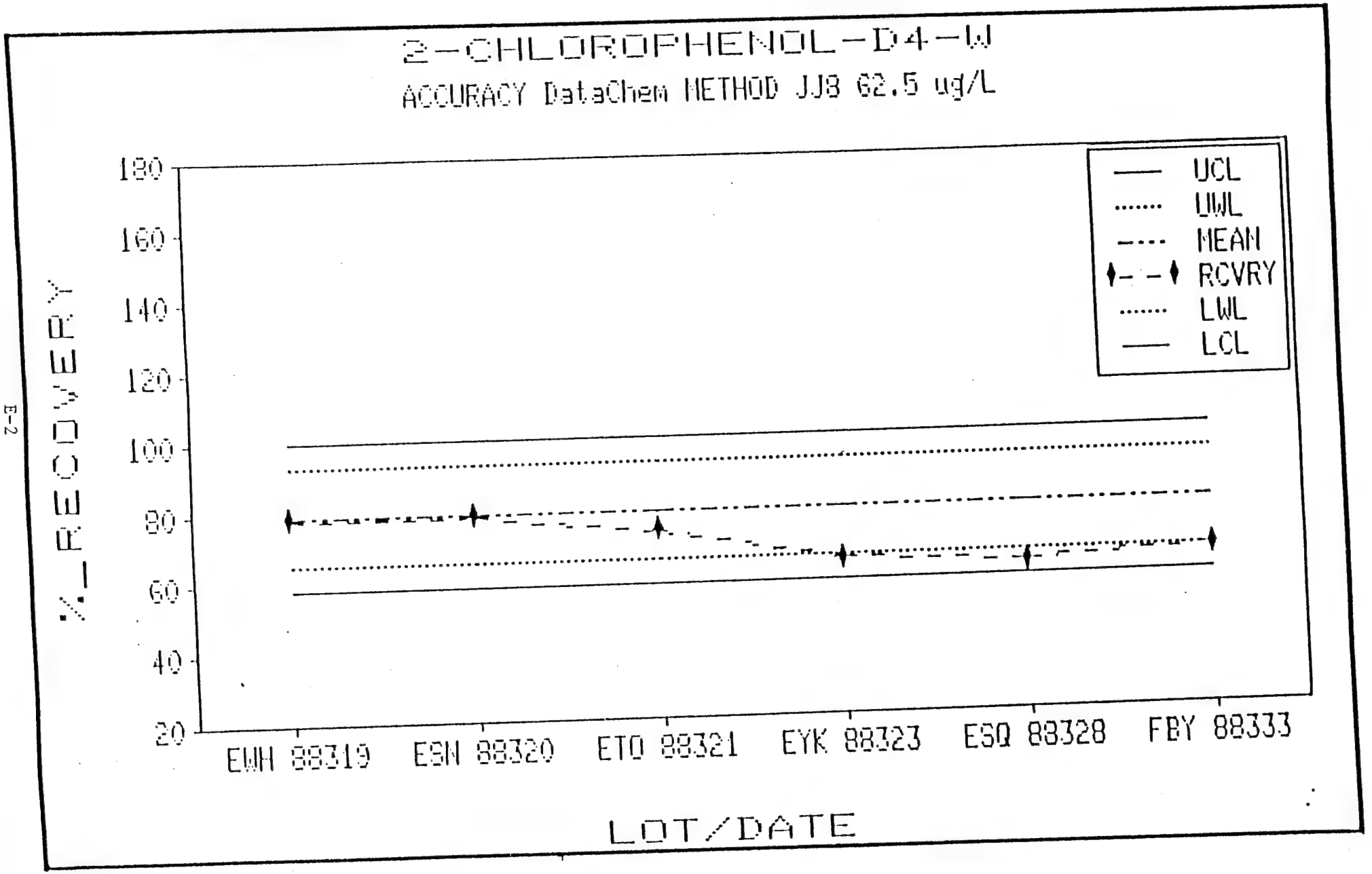
REC #	LOT	DATE	TARGET	FOUND 1	FOUND 2	RECOVERY 1	RECOVERY 2	MEAN RECOVERY
99	ERT	28-OCT-1988	0.50	0.3490	0.4167	69.80	83.34	76.57
100	ERD	29-OCT-1988	0.50	0.3990	0.3980	79.80	79.60	79.70
101	ESS	31-OCT-1988	0.50	0.4421	0.4373	88.42	87.46	87.94
102	ESZ	3-NOV-1988	0.50	0.4059	0.3521	81.18	70.42	75.80
103	ETF	3-NOV-1988	0.50	0.3577	0.3895	71.54	77.90	74.72
104	ETX	5-NOV-1988	0.50	0.3894	0.3701	77.88	74.02	75.95
105	EUT	7-NOV-1988	0.50	0.4328	0.4317	86.56	86.34	86.45
106	EWB	10-NOV-1988	0.50	0.3128	0.3176	62.56	63.52	63.04
107	EWJ	14-NOV-1988	0.50	0.3115	0.3104	62.30	62.08	62.19
108	EYA	15-NOV-1988	0.50	0.3040	0.3210	60.80	64.20	62.50
109	EYF	17-NOV-1988	0.50	0.3820	0.3891	76.40	77.82	77.11
110	EXH	18-NOV-1988	0.50	0.4615	0.4007	92.30	80.14	86.22
111	EZH	21-NOV-1988	0.50	0.4234	0.3950	84.68	79.00	81.84
112	EZD	22-NOV-1988	0.50	0.4230	0.4060	84.60	81.20	82.90
113	FAQ	30-NOV-1988	0.50	0.4006	0.4021	80.12	80.42	80.27
114	FBM	2-DEC-1988	0.50	0.4152	0.4122	83.04	82.44	82.74
115	FBS	5-DEC-1988	0.50	0.3707	0.3873	74.14	77.46	75.80
116	FDL	6-DEC-1988	0.50	0.4804	0.4641	96.08	92.82	94.45
117	FDK	12-DEC-1988	0.50	0.4110	0.4160	82.20	83.20	82.70
118	FGE	12-DEC-1988	0.50	0.4298	0.4387	85.96	87.74	86.85

MEAN RECOVERY:	82.74
ACCURACY UPPER CONTROL LIMIT:	91.53
ACCURACY LOWER CONTROL LIMIT:	73.96
AVERAGE RANGE:	4.67
PERCISION CONTROL LIMIT:	15.27

Appendix E

CLASS 1A (GC/MS) CONTROL CHART


2-CHLOROPHENOL-D4-W
PRECISION DataChem METHOD JJ8 62.5ug/L
UCL
UWL
MEAN
RANGE_%
160
140
120
100
80
60
40
20
0
RANGE_%
EWH 88319
ESN 88320
ETO 88321
EYK 88323
ESQ 88328
FBY 88333
LOT/DATE


2-CHLOROPHENOL-D4-W
ACCURACY DataChem METHOD JJ8 62.5 ug/L
% RECOVERY
180
160
140
120
100
80
60
40
20
UCL
UWL
MEAN
RCVRY
LWL
LCL
EWH 88319
ESN 88320
ETO 88321
EYK 88323
ESQ 88328
FBY 88333
LOT/DATE

2-CHLOROPHENOL-D4-W

LOW SPIKE VALUE

REC #	LOT	DATE	TARGET	ANALYTE FOUND	RECOVERY	MEAN RECOVERY
55	EWH	88319	62.500	48.1600	77.06	78.56
56	ESN	88320	62.500	50.2000	80.32	78.59
57	ETO	88321	62.500	40.2000	64.32	73.90
58	EYK	88323	62.500	29.7400	47.58	64.07
59	ESQ	88328	62.500	47.1000	75.36	62.42
60	FOY	88333	62.500	45.0700	72.11	65.02

MEAN RECOVERY:	79.36
ACCURACY UPPER CONTROL LIMIT:	100.69
ACCURACY LOWER CONTROL LIMIT:	58.03
AVERAGE RANGE:	20.85
PRECISION CONTROL LIMIT:	53.69

Appendix F

FIELD SAMPLING CHECKLIST

FIELD CHECKLIST

Field Observations

Yes__ No__ N/A__ 1. Was permission granted to enter and inspect the facility? (Required if RCRA inspection)

Yes__ No__ N/A__ 2. Is permission to enter the facility documented? If yes, where is it documented?

Yes__ No__ N/A__ 3. Were split samples offered to the facility? If yes, was the offer accepted or declined?

Yes__ No__ N/A__ 4. Is the offering of split samples recorded? If yes, where is it recorded?

Yes__ No__ N/A__ 5. If the offer to split samples was accepted, were the split samples collected? If yes, how were they identified?

Yes__ No__ N/A__ 6. Were the number, frequency, and types of field measurements and observations taken as specified in the project plan or as directed by the project coordinator? If yes, where are they recorded?

Yes__ No__ N/A__ 7. Were samples collected in the types of containers specified for each type of analysis? If no, what kind of sample containers were used?

Yes__ No__ N/A__ 8. Were samples preserved as required? If no or N/A, explain.

Yes__ No__ N/A__ 9. Were the number, frequency, and types of samples collected as specified in the project plan or as directed by the project coordinator? If no, explain why not.

Yes__ No__ N/A__ 10. Are samples packed for preservation when required (i.e., packed in ice, etc.)? If no or N/A, explain why.

Yes__ No__ N/A__ 11. Is sample custody maintained at all times? How?

FIELD CHECKLIST

Checklist for Mechanically Cored Samples

Yes__ No__ N/A__ 1. Was the rig set up at a staked and cleared borehole location?

Yes__ No__ N/A__ 2. Was the location, date, time, and other pertinent information recorded on boring log form?

Yes__ No__ N/A__ 3. Was polybutyrate core tube cut to specification and placed into the core barrel?

Yes__ No__ N/A__ 4. Was augering and coring conducted according to the following sequence: 0-1 ft, 1-4 ft, 4-5 ft, 5-9 ft, and 9-10 ft, etc.?

Yes__ No__ N/A__ 5. Was the core barrel removed from the borehole and opened at the completion of each coring interval?

Yes__ No__ N/A__ 6. Was the 12-inch section for laboratory analysis removed, capped with teflon film lined plastic caps and sealed with tape, and immediately placed in a cooler?

Yes__ No__ N/A__ 7. Were core sections which were previously etched length-wise, taped with plastic caps to prevent opening during transport to the support facility?

Yes__ No__ N/A__ 8. Were the polybutyrate lined sections marked with an arrow to the top end, the boring number, and depth interval? Was a label giving the same information as well as the project name and number, the date, and the sampler/s initials attached to the core in the sample handling trailer or at the site?

Yes__ No__ N/A__ 9. Were clean polybutyrate liners placed in a clean core barrel for each additional coring increment to be drilled?

Yes__ No__ N/A__ 10. Did the boring reach a predetermined depth or encounter the water table, whichever came first?

Yes__ No__ N/A__ 11. For trench disposal areas was the coring performed to the maximum depth of observable contamination?

Yes__ No__ N/A__ 12. Were all core sections transported to the support facility for logging and sample shipment preparation?

Yes__ No__ N/A__ 13. Was the boring stake left in the ground adjacent to the borehole and a board placed over the hole until it was grouted?

Yes__ No__ N/A__ 14. Were all boreholes greater than 1-foot in depth grouted the same day of construction and the borehole location stake placed in the grout?

Yes__ No__ N/A__ 15. Were 1-foot deep borings backfilled with native materials available adjacent to the boring?

Yes__ No__ N/A__ 16. Were the augers, and other downhole equipment decontaminated in the field prior to moving to the next borehole location upon completion of each boring?

Yes__ No__ N/A__ 17. When all borings in a specific source were completed was the drill rig initially cleaned at the source location?

Yes__ No__ N/A__ 18. Upon completion of the initial cleaning was the drill rig transported to the decontamination pad where it was thoroughly steam-cleaned before entering another source area?

Yes__ No__ N/A__ 19. Were enough augers and core barrels available so that when one set was in use a second set was being decontaminated?

Yes__ No__ N/A__ 20. At the end of the working day did all equipment, except the drill rig, and personnel proceed to the decontamination pad where decontamination procedures were initiated?

Yes__ No__ N/A__ 21. Were all bore cuttings drummed and stored while awaiting USATHAMA's directions for disposal?

FIELD CHECKLIST

Checklist for Hand Cored Samples

Yes__ No__ N/A__ 1. Was a piece of teflon film and plywood placed over the top of the polybutyrate tube and the Tube pushed or driven into the ground by hand?

Yes__ No__ N/A__ 2. Was the tube removed from the ground by shovel, the tube exterior wiped clean, the ends capped with teflon film lined plastic caps and sealed with tape?

Yes__ No__ N/A__ 3. Were the sample tubes marked with the boring number, the depth of the interval sampled, and the upward direction?

Yes__ No__ N/A__ 4. Was a label containing the same information written on the sample tube as well as the project name and number, the date and sampler's initials taped to the outside of the core?

Yes__ No__ N/A__ 5. Were cores logged and stored in a cooler with commercially available Blue Ice prior to and during transport to the support facility sampling area where they were logged for shipment?

FIELD CHECKLIST

Document Control

Yes__ No__ N/A__ 1. Have all unused and voided accountable documents been returned to the coordinator by the team members?

Yes__ No__ N/A__ 2. Were any accountable documents lost or destroyed? If yes, have document numbers of all lost or destroyed accountable documents been recorded and where are they recorded?

Yes__ No__ N/A__ 3. Are all samples identified with sample tags? If no, how are samples identified?

Yes__ No__ N/A__ 4. Are all sample tags completed (e.g., station no., location, date, time, analyses, signatures of samplers, type, preservatives, etc.)? If yes, describe types of information recorded?

Yes__ No__ N/A__ 5. Are all samples collected listed on a chain-of-custody record? If yes, describe the type of chain-of-custody record used and what information is recorded?

Yes__ No__ N/A__ 6. If used, are the sample tag numbers recorded on the chain-of-custody documents?

Yes__ No__ N/A__ 7. Does information on sample tags and chain-of-custody records match?

Yes__ No__ N/A__ 8. Does the chain-of-custody record indicate the method of sample shipment?

Yes__ No__ N/A__ 9. Is the chain-of-custody record included with the samples in the shipping container?

Yes__ No__ N/A__ 10. If used, do the sample traffic reports agree with the sample tags?

Yes__ No__ N/A__ 11. If required, has a receipt for samples been provided to the facility (required by RCRA)? Describe where offer or a receipt is documented.

Yes__ No__ N/A__ 12. If used, are blank samples identified?

Yes__ No__ N/A__ 13. If collected, are duplicate samples identified on sample tags and chain-of-custody records?

Yes__ No__ N/A__ 14. If used, are spike samples identified?

Yes__ No__ N/A__ 15. Are logbooks signed by the individual who checked out the logbook from the project coordinator?

Yes__ No__ N/A__ 16. Are logbooks dated upon receipt from the project coordinator?

Yes__ No__ N/A__ 17. Are logbooks project-specific (by logbook or by page)?

Yes__ No__ N/A__ 18. Are logbook entries dated and identified by author?

Yes__ No__ N/A__ 19. Is the facility's approval or disapproval to take photographs noted in a logbook?

Yes__ No__ N/A__ 20. Are photographs documented in logbooks (e.g., time, date, description of subject, photographer, etc.)?

Yes__ No__ N/A__ 21. If film from a self-developing camera is used, are photos matched with logbook documentation?

Yes__ No__ N/A__ 22. Are sample tag numbers recorded? If yes, describe where they are recorded?

FIELD CHECKLIST

Debriefing with Project Coordinator

Yes__ No__ N/A__ 1. Was a debriefing held with project coordinator and/or other participants?

Yes__ No__ N/A__ 2. Were any recommendations made to the project participants during the debriefing? If yes, list recommendations.